Quality Assurance Project Plan

Polychlorinated Biphenyl Sampling
In Air and Non-Porous Surfaces
For Monroe School District

Sky Valley Educational Center
351 Short Columbia Street
Monroe, Washington 98272

Effective Date: April 1, 2017

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Revision History

March 6, 2017

Revision – Revised Table 4 to reflect sample collection location as the West Pod Mezzanine.

March 24, 2017

Revisions – Incorporated Corrective Action Plan, including specific agreed to minimum sampling events and locations. Generalized document and removed specific number and location of all samples and dates of sample collection. Removed tables (2, 3, and 4) specifying sampling locations and identifying collocated air samples provided to U.S. Environmental Protection Agency (EPA). Removed references to Manchester Environmental Laboratory. Remove all reference to collocated sample collection for delivery to EPA. Revised Table 5 to Table 2.

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1.0 Purpose

This Quality Assurance Project Plan (QAPP) has been prepared by Fulcrum Environmental Consulting, Inc. (Fulcrum) to direct the collection, testing, and date evaluation of air and wipe samples from select locations in three buildings at the Sky Valley Educational Center campus, located at 351 Short Columbia in Monroe, Washington. Previous investigation identified the presence of polychlorinated biphenyl (PCB) containing lighting ballasts and caulk in the building. While ballast replacement and caulk removal have been completed, recent testing identified elevated levels of PCB in air and wipe samples collected from the building. The purpose of sampling is to evaluate the effectiveness of the PCB remediation process.

Testing is required under the Corrective Action Plan prepared by the Monroe School District (District) and agreed to by the United States Environmental Protection Agency (EPA) and Snohomish County Health District. The plan specifies the following minimum sampling by the District:

- Wipe samples will be collected from a minimum of 25% of the interior spaces where PCB containing caulk was removed to ensure that any residual PCBs are not leaching through the encapsulant layers. The District will conduct three sampling events in the first year, after epoxy encapsulant curing, December or January, and in June or July 2017. Sampling shall continue once annually in the summer until the building is renovated or demolished.

- Air samples will be collected from each interior space from which PCB caulk was removed around windows, doors or structural columns. Sampling shall be completed once per quarter beginning in August 2016.

This QAPP addresses the collection of air samples and wipe samples from discrete locations within the buildings. PCB sampling is required in the Gym Building, Pod/Library Building, Admin Building, and Annex Building. No sampling is required in the Technology Building based on the locations of PCB caulk that was identified. See Appendix A for site maps of the five site buildings including the areas of PCB caulk remediation.

2.0 Problem Definition/Background

The purpose of this sampling event is to complete quarterly sampling of air and non-porous surfaces for the presence of PCBs.

Testing will be completed for the presence of the follow PCB Aroclors:

Aroclor 1016	Aroclor 1242	Aroclor 1260
Aroclor 1221	Aroclor 1248	Aroclor 1262
Aroclor 1232	Aroclor 1254	Aroclor 1268

3.0 Project Management

The District operates the Sky Valley Educational Center campus and is the "client" for the project. The client contact is Devlin Piplic, Facilities Directors for the District.

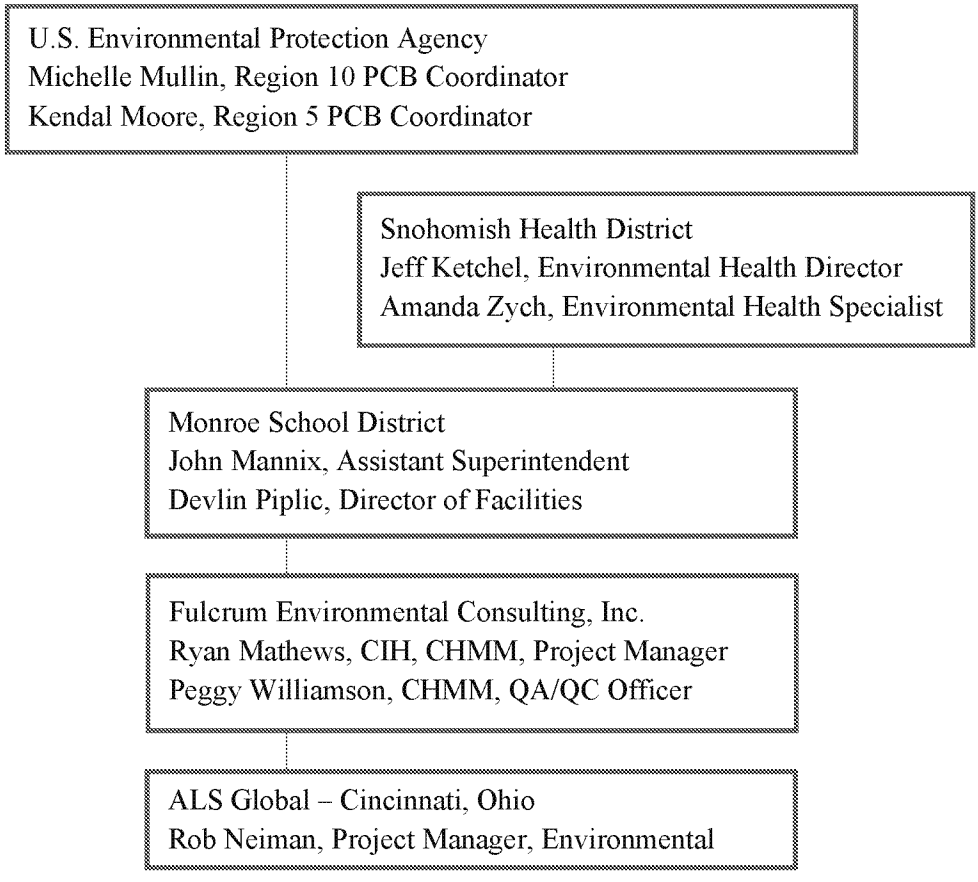
Fulcrum's Project Manager for this work is Ryan Mathews, a Certified Industrial Hygienist (CIH) and Certified Hazardous Materials Manager (CHMM).

The QA/QC Officer for this work is Peggy Williamson, CHMM, with Fulcrum.

Field tasks will be completed by an industrial hygienist with Fulcrum.

Following receipt of laboratory results and completion of initial quality assurance/quality control (QA/QC) review, Fulcrum will provide final results to the District. The District shall be responsible for dissemination of the sampling results to the EPA, Snohomish Health District representatives, and others.

4.0 Project Organization



5.0 Project/Task Description

Sampling Organization: Fulcrum Environmental Consulting, Inc.
406 North 2nd Street
Yakima, Washington 98901
Phone: 509.574.0839
Contact: Ryan Mathews

Analytical Organization: ALS Global, Cincinnati, Ohio
4388 Glendale Milford Road
Cincinnati, Ohio 45242
Phone: 513.733.5336
Contact: Rob Nieman

6.0 Quality Objectives and Criteria

The intent of the work is to generate sample results using standard sample collection and analytical methods that produce reliable laboratory results for the presence and concentration of PCBs in air and from non-porous surfaces. Analytical methods to be used are included in Table 1:

Table 1: Laboratory and Field Methodologies

Method	Method ID	Method Title
Air Sample	EPA Method TO-10a	Determination of Pesticides and Polychlorinated Biphenyls in Ambient Air Using Low Volume Polyurethane Foam (PUF) Sampling Followed by Gas Chromatographic/Multi-Detector Detection (GC/MD), January 1999.
Wipe Sample	ASTM D6661-01 [2010]	Field Collection of Organic Compounds from Surfaces Using Wipe Sampling
	40 CFR 761.123 and 130	Wipe
	EPA Method 8082A	Polychlorinated Biphenyls (PCBs) by *Gas Chromatography*, Revision 1, February 2007.

See Appendix B and C for the respective analytical methodologies.

The project laboratory will follow their standard analytical quality assurance/quality control (QA/QC) policies, practices, and procedures.

7.0 Document and Records

Samples collected from the field will be delivered to the project laboratory by commercial carrier under chain of custody. See Appendix D for a copy of an example chain of custody and a blank chain of custody.

Final analytical reports will be generated by the respective laboratory following standard laboratory practices.

Electronic versions of the reports will be emailed to the project manager in a portable document format (PDF).

An original hard copy of the report with the supporting analytical laboratory QC documentation will be kept on file at the respective laboratory. All QC documentation shall be available upon request.

Field documents will be maintained by Fulcrum's project manager.

8.0 Data Generation and Acquisition

8.1 Schedule

The project schedule will be determined as required under the Corrective Action Plan, but will generally consist of sample collection during typical period of building operation, but no or low student and staff census so as not to disrupt the learning process. For example, sampling will generally be completed during scheduled educational breaks, including spring break, winter break, summer break, etc.,

All air samples will consist of an approximate 408 minute collection period (approximate 6.8 hours). Wipe samples shall be collected during the air sampling period.

8.2 Sampling Locations & Type

Wipe sample locations shall be selected on a per event basis but will include sample collection from a minimum of 25% of the interior spaces from which PCB caulk was removed around the windows, doors, or structural members.

Air sampling locations for the project shall consist of each interior location from which PCB caulk was removed around the windows, doors, or structural members. In total 50 interior locations were identified with PCB caulk and after PCB caulking remediation, an epoxy encapsulant was applied.

Sampling locations include those areas from which the previous sampling laboratory analysis indicates the potential for PCB presence. See Appendix A for project sampling location maps.

8.3 Air Samples

Each air sample will be collected as a continuous-flow low-volume (1 to 5 liters per minute (LPM)) sample designed to collect airborne vapors on a sorbent cartridge containing polyurethane foam (PUF). The cassettes are not prepared with a pre-filter. Sampling details are provided in EPA Method TO-10a and included in Appendix B. The methodology and sample collection shall provide a method detection limit that is at or below the EPA regulatory threshold of 100 nanograms per cubic meter (ng/m^3) of air.

Sampling Train: The sampling train for collection of air samples shall consist of an air pump, a sampling cartridge with PUF media, and connecting Tygon-type flexible tubing. The sample shall be suspended above

the floor using a tripod stand with the sample collection height of between 4 and 5 feet above the floor level. The air sampling pump and the base of the tripod stand shall be placed within a 5-gallon bucket to reduce the points of contact between equipment and potentially contaminated surfaces.

Calibration: The air flow shall be calibrated prior to and following sample collection using a TSI 4046 Primary calibrator. The primary calibrator is National Institute of Standards and Technology (NIST) certified and factory calibrated annually. Calibration shall occur in-line for each sample, with the primary calibrator connected between the sample cassette and the pump.

Air Pumps: Air sampling pumps will be continuous-flow sampling pump capable of providing a constant air flow (< ±5%). Air pumps will consist of either GAST 1532 or like model high-volume pumps and/or Thomas MegaLite High Volume pumps. A total of 17 separate pumps will be used during the work to ensure prevention of air pump or stand related cross-contamination.

Sampling Cartridge and Media: Sampling cartridges shall consist of new or refurbished and laboratory certified clean cartridge constructed from a 20-millimeter (mm), inside diameter (I.D.) by 10-cm borosilicate glass tube drawn down to a 7-mm, outside diameter (O.D.), open connection for attachment to the pump by way of flexible tubing. The sorbent shall consist of a 22-mm O.D. and 7.6-centimeter (cm) long PUF and fitted under slight compression inside the cartridge. The PUF should be of the polyether type with a density of 0.0225 grams per centimeter.

8.4 Wipe Samples

Each wipe test shall be collected as provided in 40 CFR 761.130 – *Sampling Requirements*. The method is used to evaluate non-porous surfaces for comparison to the EPA threshold of 10 micrograms (mg) per 100 square centimeters (cm^2). Each wipe sample shall be laboratory prepared and consist of new cotton gauze and saturated with laboratory grade hexane. The wipes shall remain sealed in the protective container until use.

At each sample location, the inspector shall select a non-porous surface in close proximity to a transformer located within the space or, where a transformer is not located, shall select a location with visible settled dust in proximity to the former location of PCB containing caulk or below a light fixture. Where non-porous surfaces are not present, such as the concrete floor of an electrical room, select areas of concrete with an intact surface and minimal pitting or deformation.

Prior to sample collection, label the sample. Do not place the sample container upon any surfaces within the evaluation area.

Using new nitrile gloves, place a 10-cm by 10-cm disposal paper template on the surface. Remove the hexane wipe from the sampling package and immediately collect the sample using an "S" motion, turn the wipe perpendicular and complete a wiping pattern in a "N" pattern, collect settled particulate from the entirety of the area within the template. Return the sample to the sampling container, being mindful to limit contact to the sampling container. Place each sample container into an resealable bag.

8.5 Sample Labeling

Each sample shall be labeled with a unique sampling number. For the project the sample numbers shall consist of the date of collection, the building and area from which the sample was collected. Following is an example of sampling labeling following the describe practice:

e.g. 030617-POD Room F

Building designations shall be abbreviated GYM, POD, ADM, and ANX.

8.6 Field and Laboratory Blank Samples

One field blank air sample per 20 actual field samples shall be handled with all project samples and submitted to the project laboratory for analysis. The field blank sample shall be handled as with all other field samples, including the removal of the sample from the outer packaging but shall not include the opening of the cassette and the exposure of the media to air. The sample shall be packaged in a like manner and delivered with all other samples to the project laboratory.

One laboratory blank air sample per 20 actual field samples shall be delivered with other samples to the project laboratory for analysis. The laboratory blank air sample(s) shall remain in the original packaging and shall not enter the building during the project. The laboratory blank air sample shall remain in the sampler's vehicle during sample collection and shall be added to the project samples only during packaging.

One blank wipe sample shall be handled with all project samples and submitted to ALS-Cincinnati for analysis. Handling shall include the removal of the sample from the outer packaging but shall not include the opening of protective container or the exposure of the media to air or a building surface. The sample shall be packaged in a like manner and delivered with all other samples to the project laboratory.

One laboratory blank wipe sample shall remain in the original packaging and shall be delivered to the laboratory for analysis. The laboratory blank air sample shall remain in the sampler's vehicle during sample collection and shall be added to the project samples only during packaging.

8.7 Sample Handling and Custody

Air samples and wipe samples submitted for laboratory analysis will be collected by qualified staff. All samples will be transported to and from the site using insulated coolers to adhere to temperature requirements as provided in the respective methodologies. All samples will be delivered by commercial carrier under chain of custody to ALS-Cincinnati the respective analytical method preservation requirements (TO-10a for air samples and EPA SW846-8082 for wipe samples).

Certified clean cartridges do not need to be chilled when shipping and are considered clean for up to 30 days when stored in sealed containers. The sampler shall wear new nitrile gloves, changing gloves between handling of each cassette, both before and after collection.

Laboratory provided wipe media shall consist of cotton gauze wipes that that been saturated with hexane. The wipes shall be stored within 4-ounce borosilicate glass jars prior to sample collection and shall be returned to the same jar after sample collection. The sampler shall wear new nitrile gloves, changing gloves between handling each wipe.

After sample collection, all samples shall be transported at less than 4 degrees Celsius (< 4°C) by packaging with gel-type ice packets.

A summary of the sampling container, preservation requirements, and holding times is presented in Table 2:

Table 2: Sample Containers, Preservation and Holding Times

Sample Type	Preservation	Holding Time
PUF Cassette for PCBs in Air	Ship in insulated cooler at < 4°C after collection	Extract within 1 week after collection, stored at < 4°C until extracted
Hexane wipes for PCBs on Non-porous surfaces	Ship in insulated cooler at < 4°C after collection	Extract within 1 week after collection, stored at < 4°C until extracted

8.8 Equipment Decontamination

All sampling equipment shall be protected during the work to prevent contamination. At the completion of sample collection, all equipment shall be recovered and any surface that has come into contact with the building shall be decontaminated.

9.0 Data Management

Analytical data generated by ALS Global will be sent to the project manager for initial review in a PDF format. Upon completion of initial review, this data will be delivered to the District for dissemination to the U.S. EPA, Snohomish Health District, and other project partners.

ALS Global will maintain hard copies of all analytical reports, including all analytical QC measurements. Unless otherwise arranged, data generated by this project will be moved to the laboratory online database following release to the project manager.

10.0 Assessment and Oversight

Overall project assessment and oversight, including field activities, and coordination with the District will be the responsibility of the project manager. The District shall have coordination responsibilities with EPA. Data assessment/evaluation may also be provided by the project QA/QC officer. Any analytical anomalies or delays encountered during laboratory operations must be communicated to the project manager in writing (email is acceptable). The project manager will also be notified in writing of any data quality limitations that may be the result of laboratory operations.

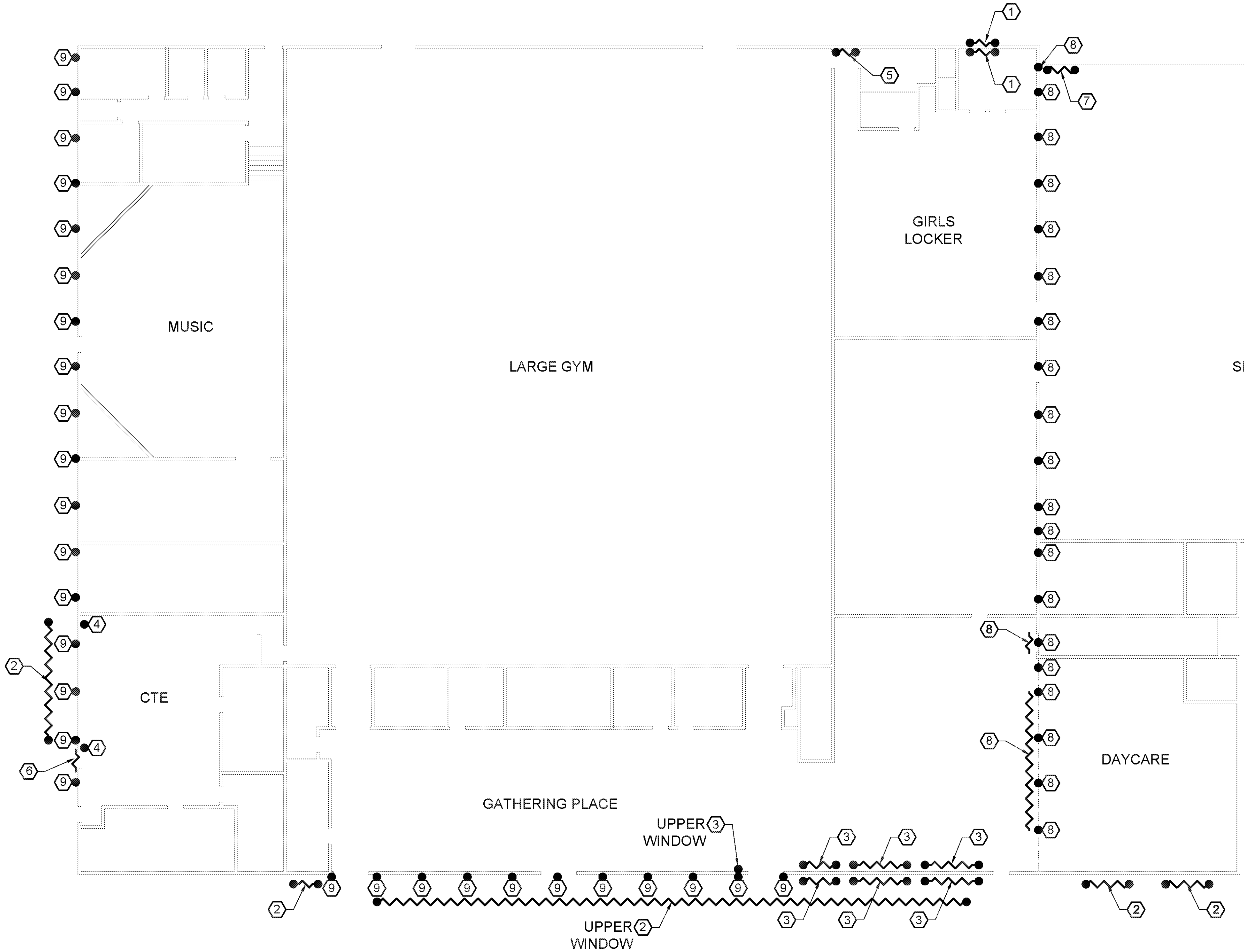
11.0 Data Validation and Usability

The project laboratory will provide standard data review, verification, and validation on all analytical data generated by this project. The extent of the data review, verification, and validation is limited to the analytical processes only. However, in the best judgment of the QA/QC officer, any data that may be inaccurate, misleading, or otherwise fails the project laboratory's quality standards due to field or sampling activities will be identified in the final analytical report.

All data verification, validation, and assessment activities for project purposes are the responsibility of the project manager.

Appendix A

Building Maps

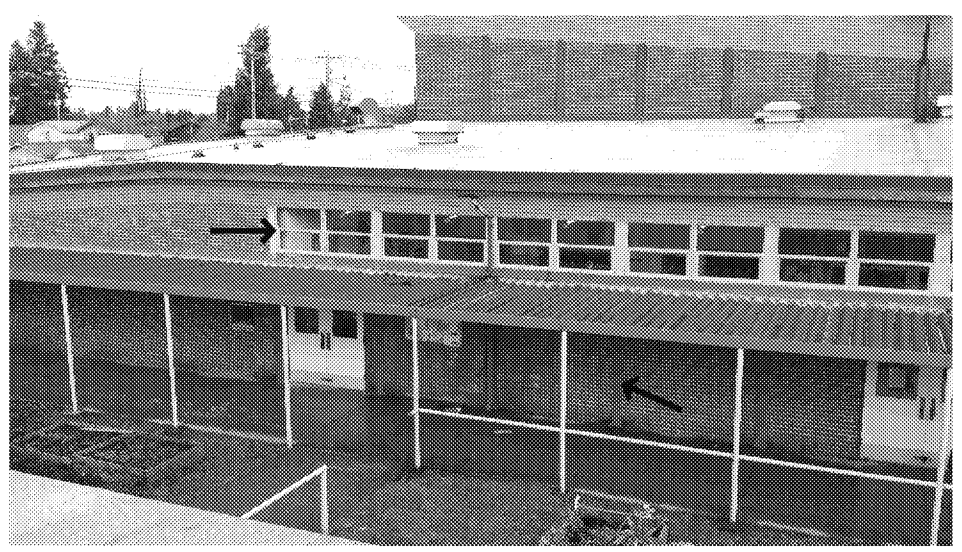


GYM BUILDING

NOT TO SCALE

LEGEND

- ● VERTICAL CAULKING RUN
- ︿︿︿ HORIZONTAL CAULKING RUNS



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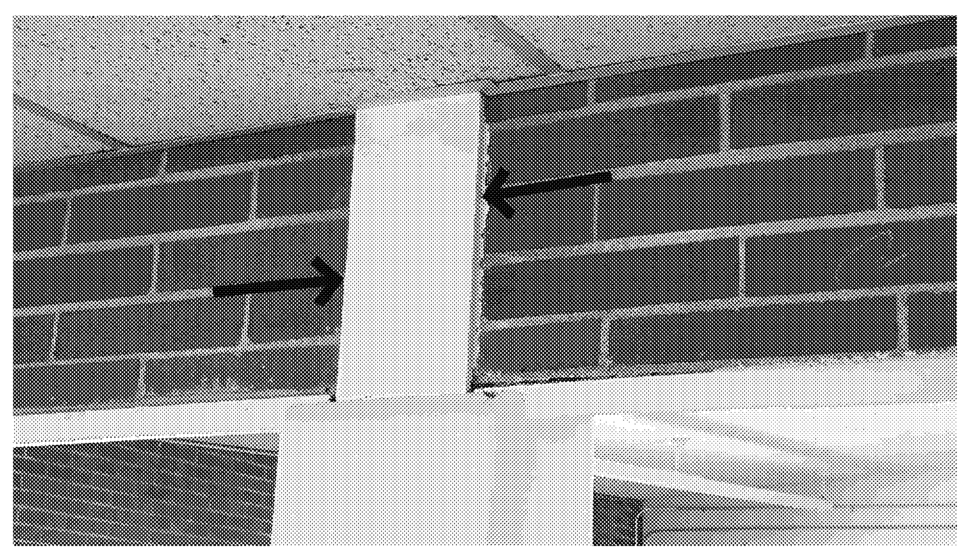

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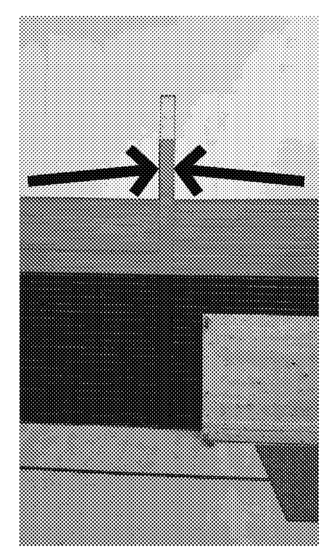

(8) **PHOTO DETAIL**

GENERAL NOTES

1. ALL ABATEMENT RELATED ACTIVITIES AT THIS PROJECT SITE SHALL COMPLY WITH DIVISION 01 AND 02 AND SPECIFICALLY SECTION 028400 PCB ACTIVITIES. CONTRACTOR TO VERIFY ALL ITEMS SHOWN, LOCATIONS AND QUANTITIES OF MATERIALS TO BE REMOVED, AND DIMENSIONS PRIOR TO REMOVAL. ANY DEVIATIONS FROM THE SPECIFICATION THAT ARE DISCOVERED BY THE CONTRACTOR SHALL BE REPORTED TO THE OWNERS REPRESENTATIVE PRIOR TO REMOVAL. THE DRAWINGS ARE FOR DIAGRAMMATIC PURPOSES ONLY. GENERAL LOCATIONS OF PCB-CONTAINING MATERIALS ARE DEPICTED DIAGRAMMATICALLY ON THE DRAWINGS. THE REMAINING MATERIAL LOCATIONS ARE DESCRIBED TEXTUALLY ON THESE DRAWINGS. QUANTITIES OF HAZARDOUS MATERIALS LISTED ON THIS SHEET ARE CONSIDERED ACCURATE TO WITHIN +/- 10%. THE CONTRACTOR SHALL PROVIDE ALL LABOR, MATERIALS, EQUIPMENT AND PERMITS FOR THE REMOVAL AND DISPOSAL OF THE QUANTITIES OF HAZARDOUS MATERIALS PROVIDED PLUS AN ADDITIONAL 10%. THE CONTRACTOR WILL BE COMPENSATED FOR QUANTITIES WHICH ARE GREATER THAN 110% OF THE TOTAL AND THE OWNER WILL DEDUCT FROM THE CONTRACT SUM QUANTITIES THAT ARE 90% OR LESS OF THE TOTAL.
2. REMOVAL OF HAZARDOUS MATERIALS MAY COMPROMISE THE SECURITY OF THE SITE. THE CONTRACTOR IS FULLY RESPONSIBLE FOR MAINTAINING SITE SECURITY AND PUBLIC SAFETY THROUGHOUT THE PROJECT. SEE SPECIFICATIONS REGARDING SECURITY AND PUBLIC SAFETY.
3. ABATEMENT CONTRACTOR TO COORDINATE ALL ACTIVITIES WITH ALL OTHER ONSITE WORK INCLUDING, BUT NOT LIMITED TO: SCHEDULE, ACCESS, STAGING, ETC. ABATEMENT CONTRACTOR TO REPORT LOCATIONS AND QUANTITIES OF ALL HAZARDOUS MATERIALS TO BE REMOVED, TO THE OWNERS REPRESENTATIVE PRIOR TO ABATEMENT/DEMOLITION.
4. THE CONTRACTOR SHALL REMOVE ALL ACCESSIBLE CAULKING IN ALL AREAS WITHOUT PERFORMING DEMOLITION OF BUILDING COMPONENTS.

KEY NOTES

(1) REMOVE APPROX. 20 LF OF PCB-CONTAINING CAULKING LOCATED ON THE EXTERIOR AND INTERIOR METAL WINDOW FRAME ON THE GIRLS LOCKER ROOM NORTH PERIMETER WINDOW AS SHOWN.

(2) REMOVE APPROX. 300 LF OF PCB-CONTAINING CAULKING ON THE EXTERIOR METAL WINDOW FRAMES ON ALL WINDOWS AT THE SOUTH AND WEST ELEVATIONS OF THE LARGE GYM BUILDING AS SHOWN. THIS INCLUDES CAULKING THAT EXISTS AROUND EACH WINDOW INFILL PANEL METAL FRAME TRANSITION ON THE WEST ELEVATION. THESE INFILL PANELS ARE CEMENT ASBESTOS BOARD.

(3) REMOVE APPROX. 40 LF OF PCB-CONTAINING CAULKING LOCATED ON INTERIOR SIDE OF THE THREE LOWER WINDOWS AND THE UPPER WINDOW BANK EAST VERTICAL IN THE GATHERING PLACE AS SHOWN.

(4) REMOVE APPROX. 10 LF OF PCB-CONTAINING OF CAULK ON INTERIOR WINDOW FRAME VERTICALS IN THE CTE ROOM AS SHOWN.

(5) REMOVE APPROX. 18 LF OF PCB-CONTAINING OF CAULK ON INTERIOR SIDE OF NORTH EXTERIOR GIRLS LOCKER ENTRY DOOR AS SHOWN.

(6) REMOVE APPROX. 18 LF OF PCB-CONTAINING OF CAULK ON EXTERIOR SIDE OF NORTH CTE ENTRY DOOR AS SHOWN.

(7) REMOVE APPROX. 18 LF OF PCB-CONTAINING OF CAULK ON THE INTERIOR SIDE OF THE NORTHWEST PERIMETER ENTRY DOOR FRAME OF THE SMALL GYM AS SHOWN.

(8) REMOVE APPROX. 500 LF OF PCB-CONTAINING CAULKING LOCATED ON ALL VERTICAL STRUCTURAL METAL COLUMN TRANSITIONS THROUGHOUT THE EAST ELEVATION OF THE LARGE GYM AS SHOWN. THIS INCLUDES THE REMOVAL OF ALL CAULKING ON THE INTERIOR DEMISING WALL METAL BEAMS (VERTICAL AND HORIZONTAL) BETWEEN THE DAYCARE AND THE GATHERING PLACE/CAFETERIA AS SHOWN. THE CAULKING IS HEAVILY PAINTED THROUGHOUT THE WORK SCOPE AREA.

(9) REMOVE APPROX. 780 LF OF PCB-CONTAINING CAULKING LOCATED ON ALL EXTERIOR VERTICAL STRUCTURAL METAL COLUMN TRANSITIONS THROUGHOUT THE LOWER WEST AND SOUTH ELEVATIONS OF THE LARGE GYM BUILDING AS SHOWN.

NOT TO SCALE

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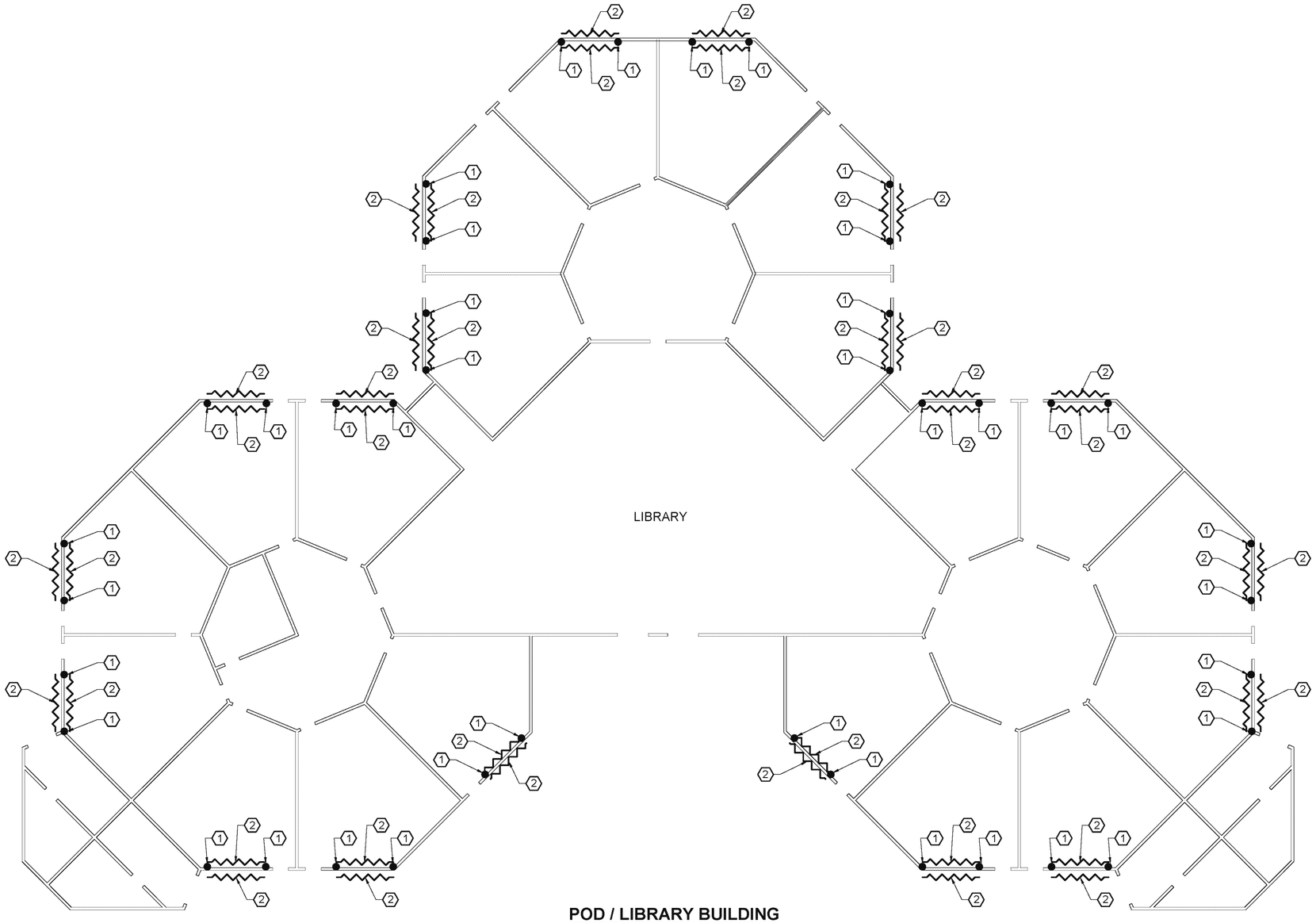
MONROE SCHOOL DISTRICT

GYM BUILDING CAULKING ABATEMENT PLAN

SKY VALLEY EDUCATIONAL CENTER

SKY VALLEY EDUCATIONAL CENTER
351 SHORT COLUMBIA STREET
MONROE, WASHINGTON

PROJECT:	41373.000
DRAWN:	JHD
CHECKED:	GM
DATE:	JUNE 2016
DWG NO.	SHEET NO.
HM1	1 OF 5



POD / LIBRARY BUILDING

NOT TO SCALE

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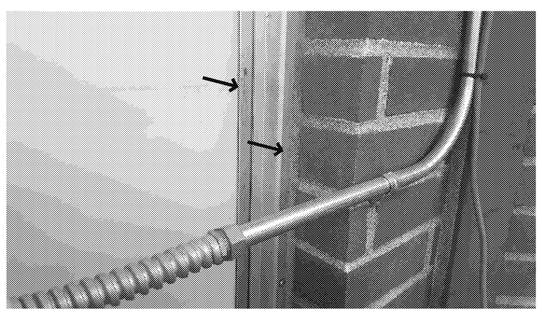
KEY NOTES

(1) REMOVE APPROX. 500 LF OF PCB-CONTAINING CAULKING LOCATED ON INTERIOR PERIMETER METAL WINDOW FRAME TO BRICK TRANSITION VERTICALS IN EACH OF CLASSROOMS 1-20 AS SHOWN.

(2) REMOVE APPROX. 1,400 LF OF PCB-CONTAINING CAULKING ON THE INTERIOR AND EXTERIOR SIDES OF THE CEMENT ASBESTOS BOARD (CAB) WINDOW INFILL PANELS. THE CAULKING FILLS THE GAP BETWEEN THE METAL WINDOW FRAME AND CAB TRANSITION IN EACH OF CLASSROOMS 1-20 AS SHOWN.

LEGEND

- VERTICAL CAULKING RUN
- HORIZONTAL CAULKING RUN



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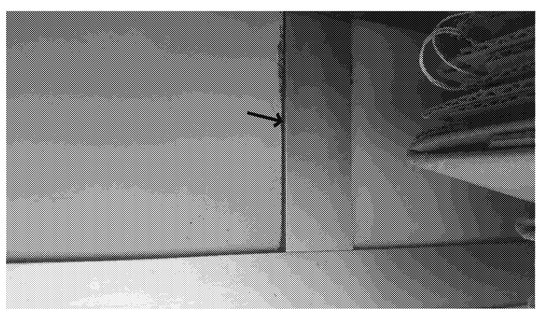

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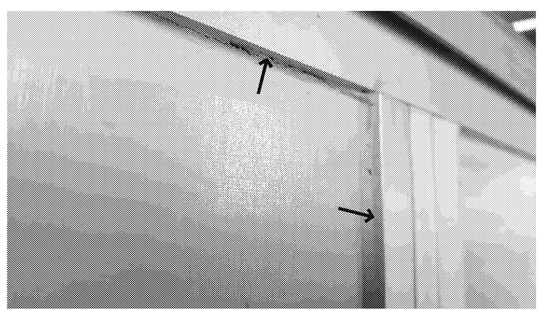

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MONROE SCHOOL DISTRICT

POD/LIBRBAY BUILDING CAULKING ABATEMENT PLAN

SKY VALLEY EDUCATIONAL CENTER

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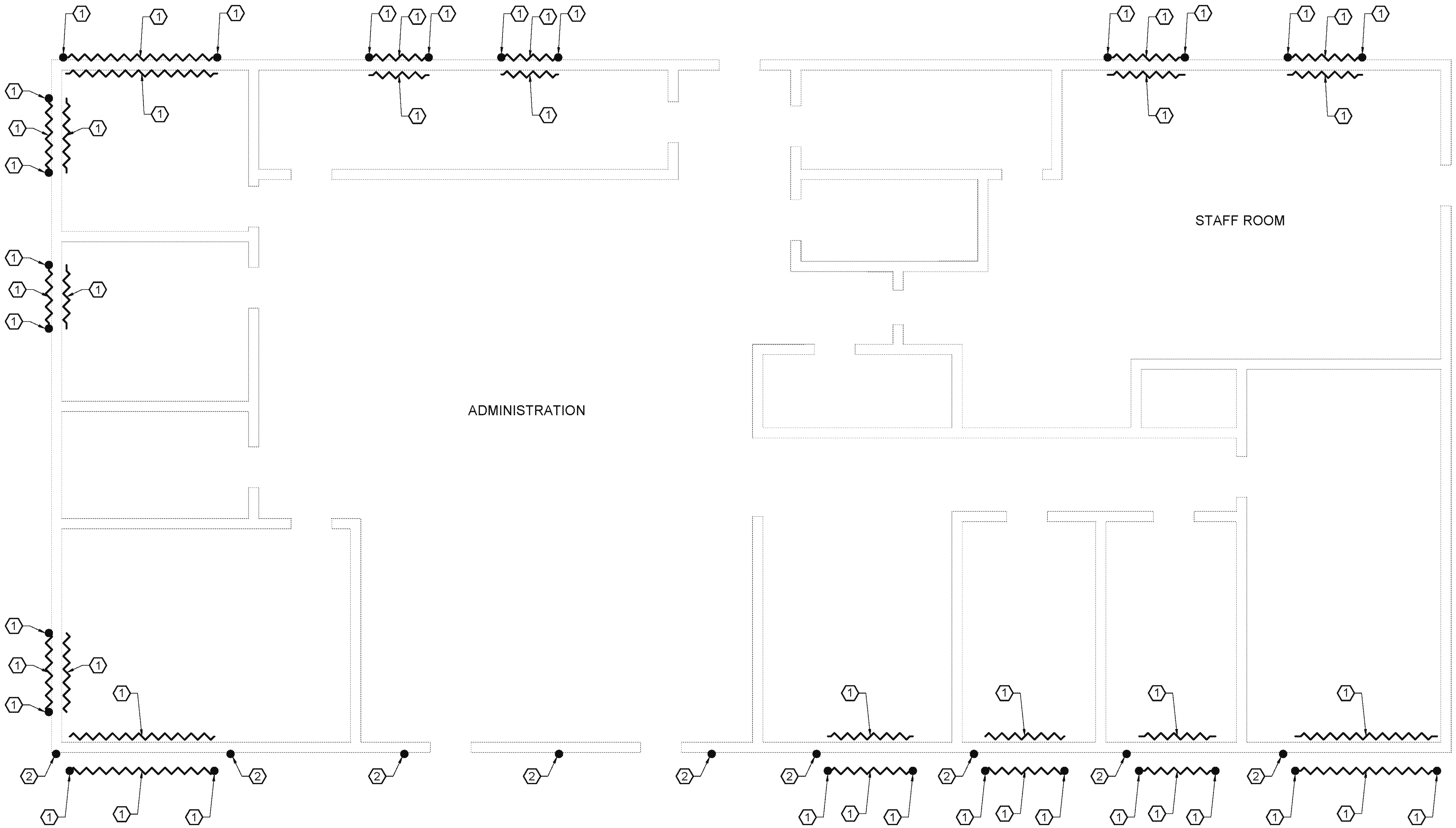
PROJECT:	41373.000
DRAWN:	JHD
CHECKED:	GM
DATE:	JUNE 2016
DWG NO.	SHEET NO.
HM2	2 OF 5



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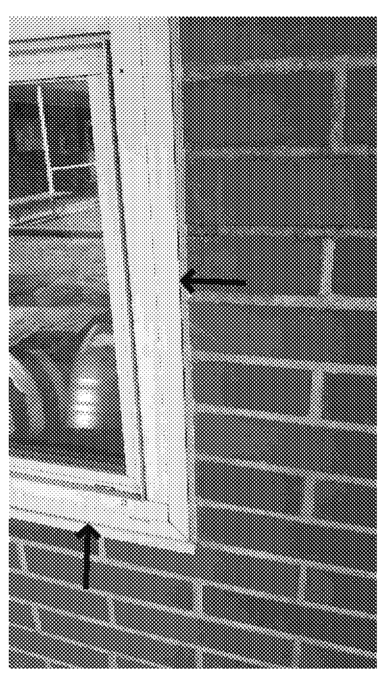

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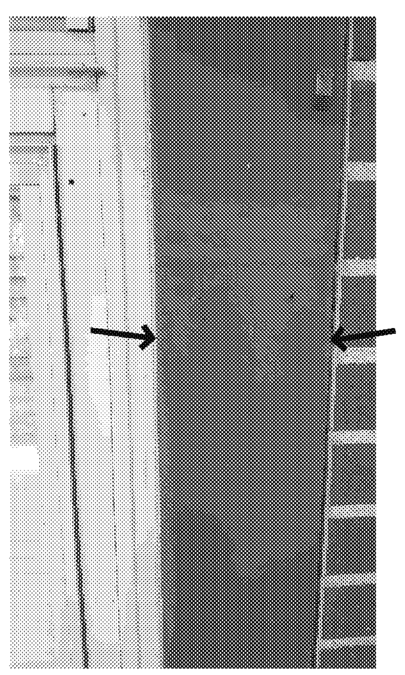

②PHOTO DETAIL

● Air Testing Sample

W Wipe Test Sample

NOT TO SCALE

GENERAL NOTES

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4. THE CONTRACTOR SHALL REMOVE ALL ACCESSIBLE CAULKING IN ALL AREAS WITHOUT PERFORMING DEMOLITION OF BUILDING COMPONENTS.

KEY NOTES

① REMOVE APPROX. 400 LF OF PCB-CONTAINING CAULKING LOCATED ON ALL INTERIOR METAL WINDOW SILL TRANSITIONS AND ALL EXTERIOR METAL WINDOW FRAME TRANSITIONS THROUGHOUT THE ADMINISTRATION BUILDING AS SHOWN.

② REMOVE APPROX. 175 LF OF PCB-CONTAINING CAULKING LOCATED ON ALL EXTERIOR VERTICAL STRUCTURAL METAL BEAM TRANSITIONS THROUGHOUT THE ADMINISTRATION BUILDING AS SHOWN.

LEGEND

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〰 HORIZONTAL CAULKING RUN

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PROJECT:	41373.000
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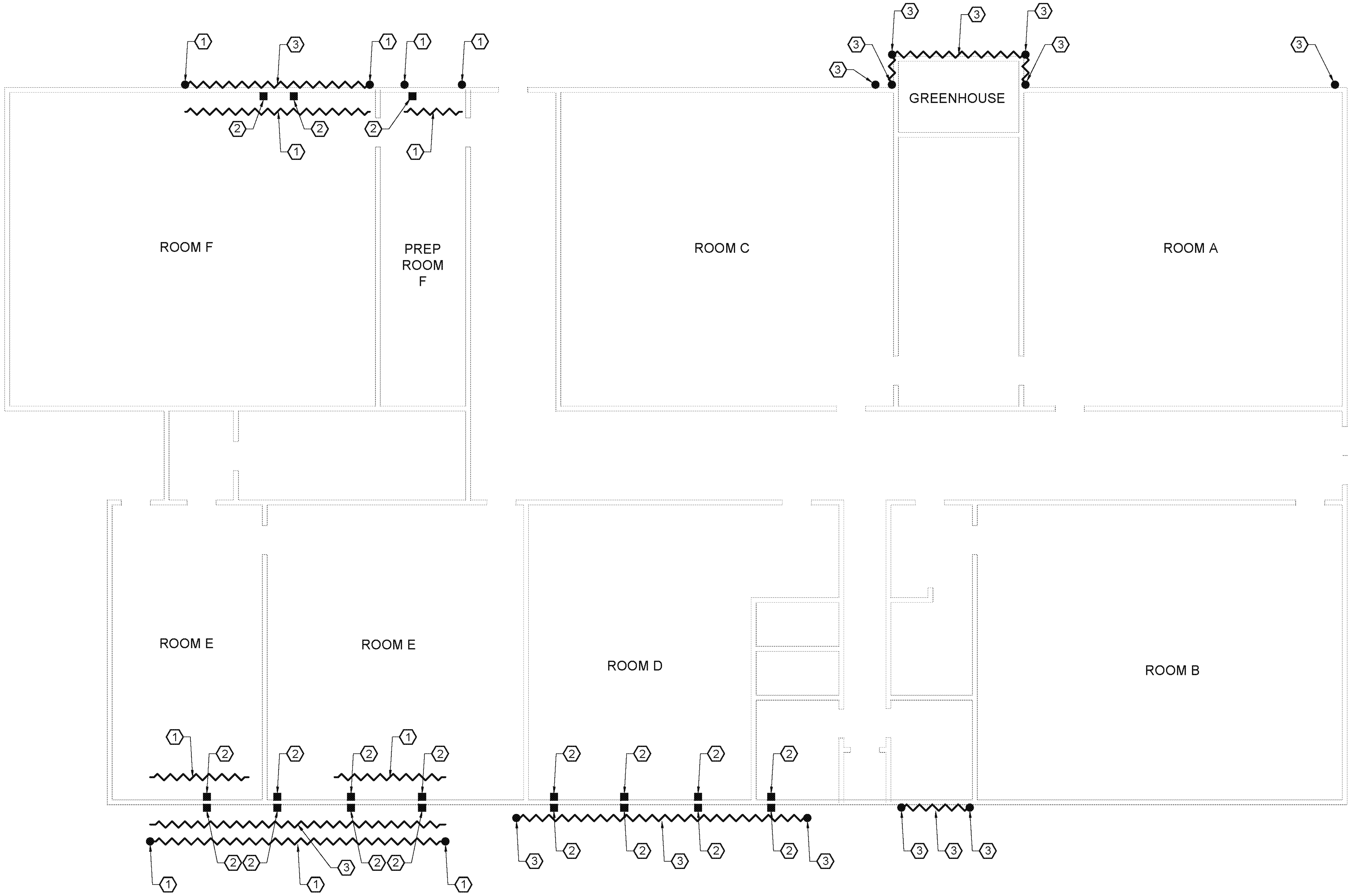
CHECKED:	GM
DATE:	JUNE 2016
DWG NO. HM3	SHEET NO. 3 OF 5



ANNEX BUILDING

NOT TO SCALE

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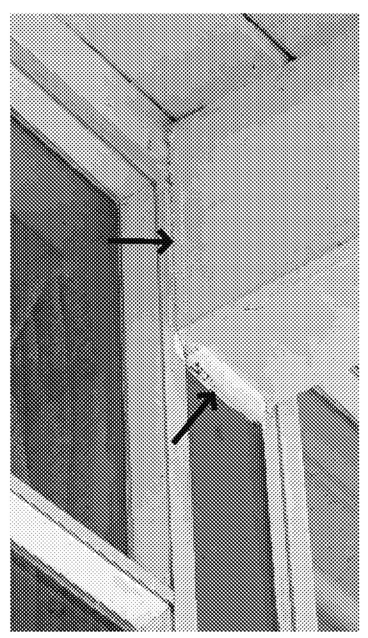

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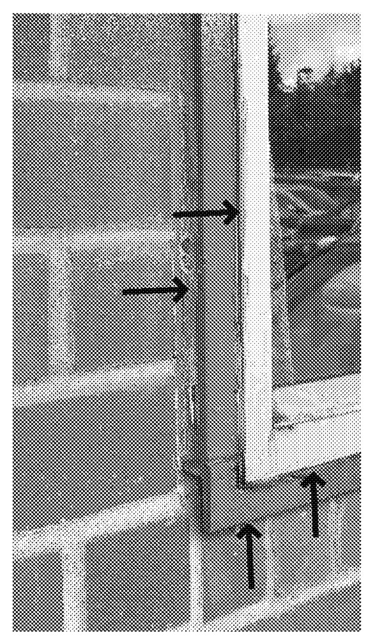

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4. THE CONTRACTOR SHALL REMOVE ALL ACCESSIBLE CAULKING IN ALL AREAS WITHOUT PERFORMING DEMOLITION OF BUILDING COMPONENTS.

KEY NOTES

(1) REMOVE APPROX. 200 LF OF PCB-CONTAINING CAULKING LOCATED ON INTERIOR PERIMETER METAL WINDOW FRAME TRANSITIONS. THIS INCLUDES REMOVAL OF CAULKING WHICH EXISTS ON EXTERIOR METAL WINDOW FRAME TRANSITIONS ON THE NORTH AND SOUTH BUILDING ELEVATION WINDOWS AS SHOWN.

(2) REMOVE APPROX. 80 LF OF PCB-CONTAINING CAULKING ON WOOD CEILING/SOFFIT BEAMS AT PERIMETER WALL/CEILING TRANSITIONS IN ROOMS E, F AND PREP ROOM F AS SHOWN.

(3) REMOVE APPROX. 300 LF OF PCB AND ASBESTOS-CONTAINING TAN CAULKING LOCATED ON VARIOUS VERTICAL AND HORIZONTAL METAL WINDOW FRAME TRANSITIONS ON THE NORTH AND SOUTH ELEVATIONS OF THE ANNEX BUILDING AS SHOWN.

LEGEND

- ● VERTICAL CAULKING RUN
- ■ CAULKING ON BEAM
- ∿∿∿ HORIZONTAL CAULKING RUN

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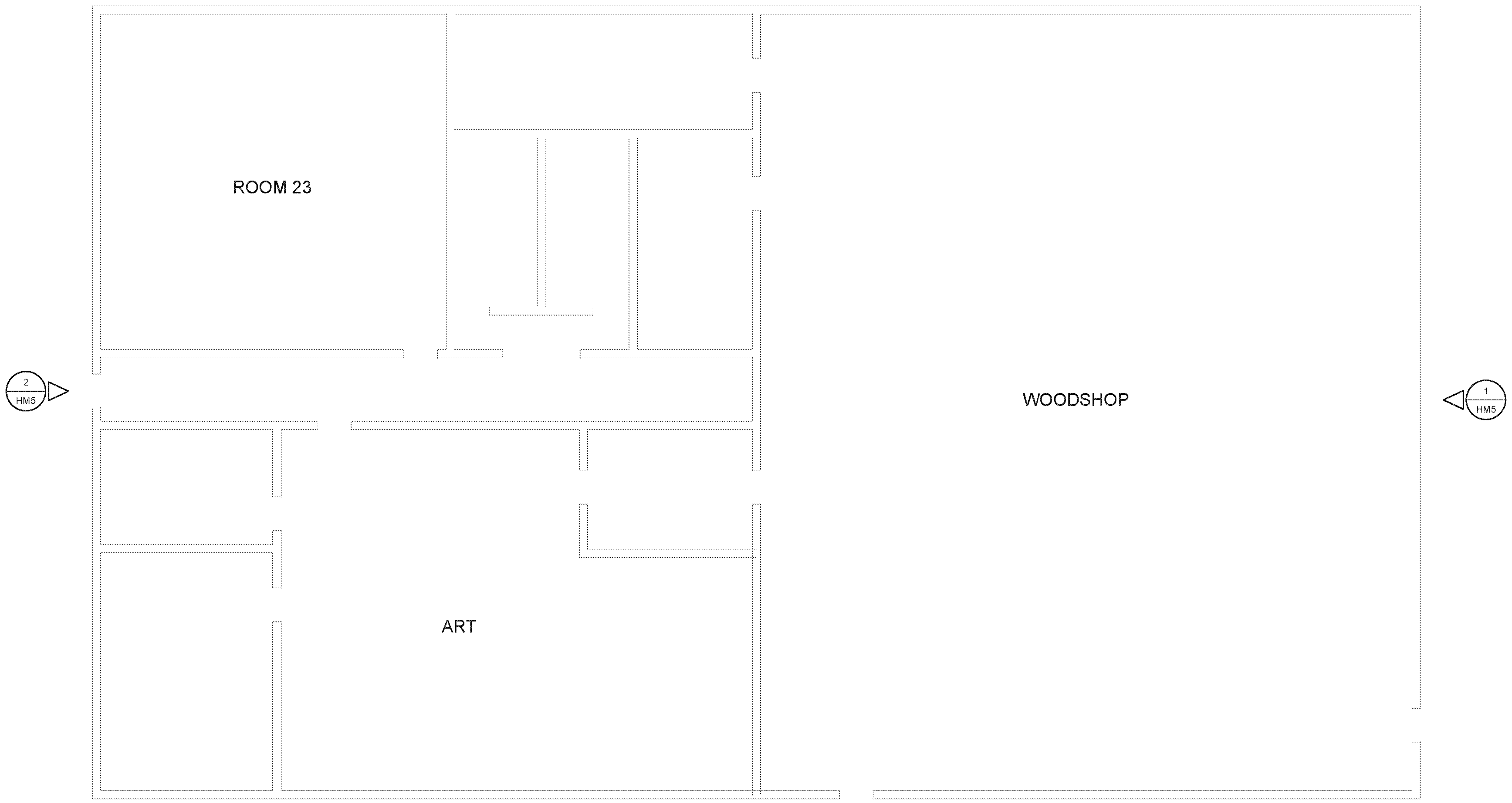
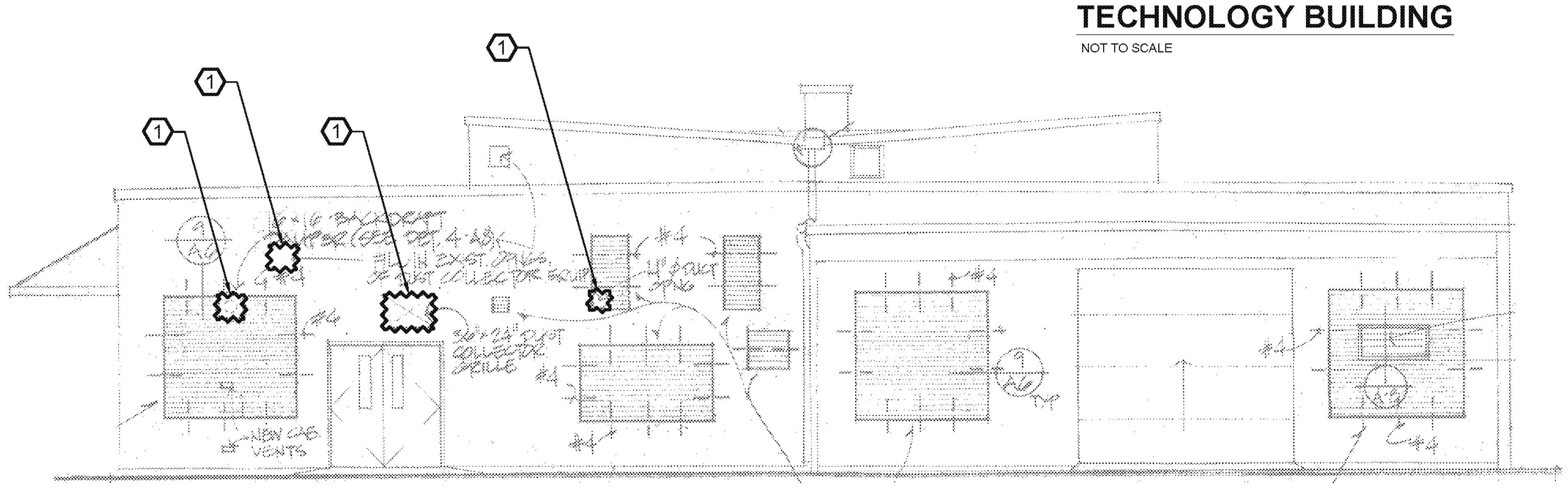
DATE:	JUNE 2016
DWG NO.: HM4	SHEET NO.: 4 OF 5



TECHNOLOGY BUILDING
NOT TO SCALE



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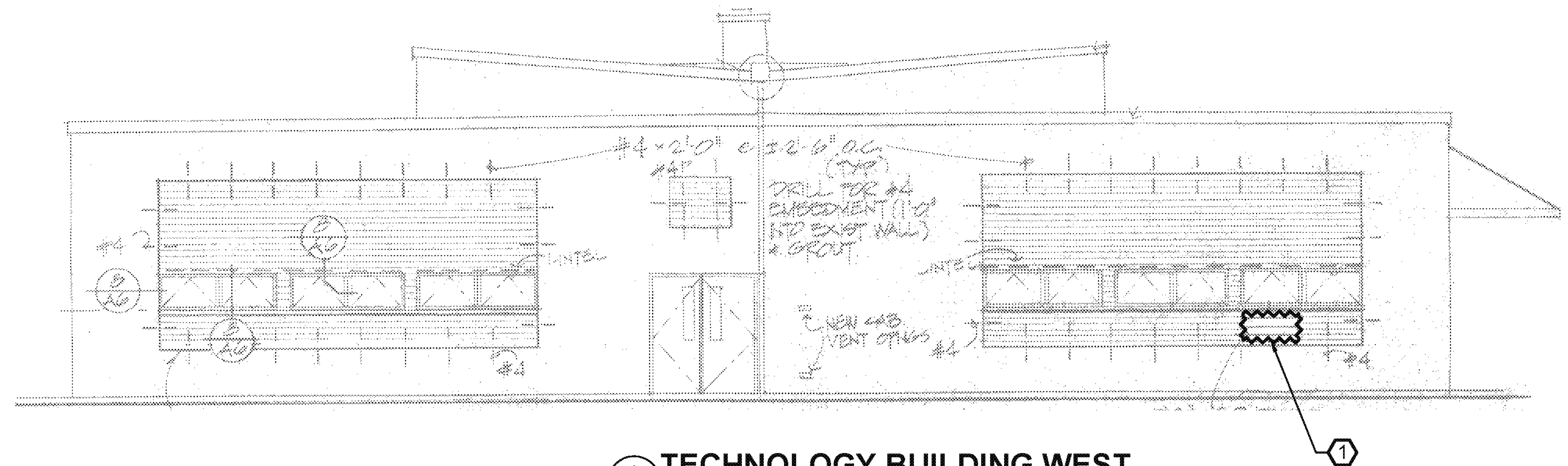

2 HM5 **TECHNOLOGY BUILDING WEST**
NOT TO SCALE

(1) **PHOTO DETAIL**

(1) **PHOTO DETAIL**

NOT TO SCALE

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4. THE CONTRACTOR SHALL REMOVE ALL ACCESSIBLE CAULKING IN ALL AREAS WITHOUT PERFORMING DEMOLITION OF BUILDING COMPONENTS.

KEY NOTES

(1) REMOVE APPROX. 60 LF OF PCB-CONTAINING CAULKING LOCATED ON EXTERIOR LOUVERS, VENTS AND DUCTING ON METAL TRANSITIONS ON THE WEST AND EAST EXTERIOR ELEVATIONS OF THE TECHNOLOGY BUILDING AS SHOWN.

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Appendix B

TO-10a Methodology

EPA/625/R-96/010b

Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air

Second Edition

Compendium Method TO-10A

Determination Of Pesticides And Polychlorinated Biphenyls In Ambient Air Using Low Volume Polyurethane Foam (PUF) Sampling Followed By Gas Chromatographic/Multi-Detector Detection (GC/MD)

Center for Environmental Research Information
Office of Research and Development
U.S. Environmental Protection Agency
Cincinnati, OH 45268

January 1999

Method TO-10A
Acknowledgements

This Method was prepared for publication in the *Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Second Edition* (EPA/625/R-96/010b), which was prepared under Contract No. 68-C3-0315, WA No. 3-10, by Midwest Research Institute (MRI), as a subcontractor to Eastern Research Group, Inc. (ERG), and under the sponsorship of the U.S. Environmental Protection Agency (EPA). Justice A. Manning, John Burckle, and Scott R. Hedges, Center for Environmental Research Information (CERI), and Frank F. McElroy, National Exposure Research Laboratory (NERL), all in the EPA Office of Research and Development (ORD), were responsible for overseeing the preparation of this method. Additional support was provided by other members of the Compendia Workgroup, which include:

- John Burckle, U.S. EPA, ORD, Cincinnati, OH
- James L. Cheney, Corps of Engineers, Omaha, NB
- Michael Davis, U.S. EPA, Region 7, KC, KS
- Joseph B. Elkins Jr., U.S. EPA, OAQPS, RTP, NC
- Robert G. Lewis, U.S. EPA, NERL, RTP, NC
- Justice A. Manning, U.S. EPA, ORD, Cincinnati, OH
- William A. McClenny, U.S. EPA, NERL, RTP, NC
- Frank F. McElroy, U.S. EPA, NERL, RTP, NC
- Heidi Schultz, ERG, Lexington, MA
- William T. "Jerry" Winberry, Jr., EnviroTech Solutions, Cary, NC

Method TO-10 was originally published in March of 1989 as one of a series of peer reviewed methods in the second supplement to *"Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air,"* EPA 600/4-89-018. In an effort to keep these methods consistent with current technology, Method TO-10 has been revised and updated as Method TO-10A in this Compendium to incorporate new or improved sampling and analytical technologies. In addition, this method incorporates ASTM Method D 4861-94, *Standard Practice for Sampling and Analysis of Pesticides and Polychlorinated Biphenyls in Air.*

This Method is the result of the efforts of many individuals. Gratitude goes to each person involved in the preparation and review of this methodology.

Author(s)
- Robert G. Lewis, U.S. EPA, NERL, RTP, NC

Peer Reviewers
- William T. "Jerry" Winberry, Jr., EnviroTech Solutions, Cary, NC
- Irene D. DeGraff, Supelco, Bellefonte, PA
- Lauren Drees, U.S. EPA, NRMRL, Cincinnati, OH

Finally, recognition is given to Frances Beyer, Lynn Kaufman, Debbie Bond, Cathy Whitaker, and Kathy Johnson of Midwest Research Institute's Administrative Services staff whose dedication and persistence during the development of this manuscript has enabled it's production.

DISCLAIMER

METHOD TO-10A

Determination Of Pesticides And Polychlorinated Biphenyls In Ambient Air Using Low Volume Polyurethane Foam (PUF) Sampling Followed By Gas Chromatographic/Multi-Detector Detection (GC/MD)

TABLE OF CONTENTS

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METHOD TO-10A

Determination Of Pesticides And Polychlorinated Biphenyls In Ambient Air Using Low Volume Polyurethane Foam (PUF) Sampling Followed By Gas Chromatographic/Multi-Detector (GC/MD) Detection

1. Scope

1.1 This document describes a method for sampling and analysis of a variety of common pesticides and for polychlorinated biphenyls (PCBs) in ambient air. The procedure is based on the adsorption of chemicals from ambient air on polyurethane foam (PUF) or a combination of PUF and granular sorbent using a low volume sampler.

1.2 The low volume PUF sampling procedure is applicable to multicomponent atmospheres containing common pesticide concentrations from 0.001 to 50 $\mu g/m^3$ over 4- to 24-hour sampling periods. The limits of detection will depend on the nature of the analyte and the length of the sampling period.

1.3 Specific compounds for which the method has been employed are listed in Table 1. The analytical methodology described in Compendium Method TO-10A is currently employed by laboratories throughout the U.S. The sampling methodology has been formulated to meet the needs of common pesticide and PCB sampling in ambient air.

1.4 Compendium Method TO-10 was originally published in 1989. The method was further modified for indoor air application in 1990. In an effort to keep the method consistent with current technology, Compendium Method TO-10 has incorporated ASTM Method D4861-94 (1) and is published here as Compendium Method TO-10A.

2. Summary of Method

2.1 A low-volume (1 to 5 L/minute) sample is used to collect vapors on a sorbent cartridge containing PUF or PUF in combination with another solid sorbent. Airborne particles may also be collected, but the sampling efficiency is not known (2).

2.2 Pesticides and other chemicals are extracted from the sorbent cartridge with 5 percent diethyl ether in hexane and determined by gas chromatography coupled with an electron capture detector (ECD), nitrogen-phosphorus detector (NPD), flame photometric detector (FPD), Hall electrolytic conductivity detector (HECD), or a mass spectrometer (MS). For common pesticides, high performance liquid chromatography (HPLC) coupled with an ultraviolet (UV) detector or electrochemical detector may be preferable. This method describes the use of an electron capture detector.

2.3 Interferences resulting from analytes having similar retention times during GC analysis are resolved by improving the resolution or separation, such as by changing the chromatographic column or operating parameters, or by fractionating the sample by column chromatography.

3. Significance

3.1 Pesticide usage and environmental distribution are common to rural and urban areas of the United States. The application of pesticides can cause potential adverse health effects to humans by contaminating soil, water, air, plants, and animal life. However, human exposure to PCBs continues to be a problem because of their presence in the environment.

3.2 Many pesticides and PCBs exhibit bioaccumulative, chronic health effects; therefore, monitoring the presence of these compounds in ambient air is of great importance.

3.3 Use of a portable, low volume PUF sampling system allows the user flexibility in locating the apparatus. The user can place the apparatus in a stationary or mobile location. The portable sampling apparatus may be positioned in a vertical or horizontal stationary location (if necessary, accompanied with supporting structure). Mobile positioning of the system can be accomplished by attaching the apparatus to a person to test air in the individual's breathing zone.

3.4 Moreover, this method has been successfully applied to measurement of common pesticides in outdoor air, indoor air and for personal respiratory exposure monitoring (3).

4. Applicable Documents

4.1 ASTM Standards

- D1356 *Definition of Terms Relating to Atmospheric Sampling and Analysis*
- D4861-94 *Standard Practice for Sampling and Analysis of Pesticides and Polychlorinated Biphenyls in Air*
- E260 *Recommended Practice for General Gas Chromatography Procedures*
- E355 *Practice for Gas Chromatography Terms and Relationships*
- D3686 *Practice for Sampling Atmospheres to Collect Organic Compound Vapors (Activated Charcoal Tube Adsorption Method*
- D3687 *Practice for Analysis of Organic Compound Vapors Collected by the Activated Charcoal Tube Adsorption*
- D4185 *Practice for Measurement of Metals in Workplace Atmosphere by Atomic Absorption Spectrophotometry*

4.2 EPA Documents

- *Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air: Method TO-10, Second Supplement,* U. S. Environmental Protection Agency, EPA 600/4-89-018, March 1989.
- *Manual of Analytical Methods for Determination of Pesticides in Humans and Environmental Standards*, U. S. Environmental Protection Agency, EPA 600/8-80-038, June 1980.
- *Compendium of Methods for the Determination of Air Pollutants in Indoor Air: Method IP-8,* U. S. Environmental Protection Agency, EPA 600/4-90-010, May 1990.

4.3 Other Documents

- Code of Federal Regulations, Title 40, Part 136, Method 604

5. Definitions

[Note: Definitions used in this document and in any user-prepared Standard operating procedures (SOPs) should be consistent with ASTM D1356, E260, and E355. All abbreviations and symbols are defined within this document at point of use.]

5.1 Sampling efficiency (SE)-ability of the sampling medium to trap analytes of interest. The percentage of the analyte of interest collected and retained by the sampling medium when it is introduced as a vapor in air or nitrogen into the air sampler and the sampler is operated under normal conditions for a period of time equal to or greater than that required for the intended use is indicated by %SE.

5.2 Retention efficiency (RE)-ability of sampling medium to retain a compound added (spiked) to it in liquid solution.

5.3 Static retention efficiency-ability of the sampling medium to retain the solution spike when the sample cartridge is stored under clean, quiescent conditions for the duration of the test period.

5.4 Dynamic retention efficiency (RE_d)-ability of the sampling medium to retain the solution spike when air or nitrogen is drawn through the sampling cartridge under normal operating conditions for the duration of the test period. The dynamic RE is normally equal to or less than the SE.

5.5 Retention time (RT)-time to elute a specific chemical from a chromatographic column, for a specific carrier gas flow rate, measured from the time the chemical is injected into the gas stream until it appears at the detector.

5.6 Relative retention time (RRT)-a rate of RTs for two chemicals for the same chromatographic column and carrier gas flow rate, where the denominator represents a reference chemical.

5.7 Surrogate standard-a chemically inert compound (not expected to occur in the environmental sample) that is added to each sample, blank, and matrix-spiked sample before extraction and analysis. The recovery of the surrogate standard is used to monitor unusual matrix effects, gross sample processing errors, etc. Surrogate recovery is evaluated for acceptance by determining whether the measured concentration falls within acceptable limits.

6. Interferences

6.1 Any gas or liquid chromatographic separation of complex mixtures of organic chemicals is subject to serious interference problems due to coelution of two or more compounds. The use of capillary or microbore columns with superior resolution or two or more columns of different polarity will frequently eliminate these problems. In addition, selectivity may be further enhanced by use of a MS operated in the selected ion monitoring (SIM) mode as the GC detector. In this mode, co-eluting compounds can often be determined.

6.2 The ECD responds to a wide variety of organic compounds. It is likely that such compounds will be encountered as interferences during GC/ECD analysis. The NPD, FPD, and HECD detectors are element specific, but are still subject to interferences. UV detectors for HPLC are nearly universal, and the electrochemical detector may also respond to a variety of chemicals. Mass spectrometric analyses will generally provide positive identification of specific compounds.

6.3 PCBs and certain organochlorine pesticides (e.g., chlordane) are complex mixtures of individual compounds which can cause difficulty in accurately quantifying a particular formulation in a multiple component mixture. PCBs may interfere with the determination of pesticides.

6.4 Contamination of glassware and sampling apparatus with traces of pesticides or PCBs can be a major source of error, particularly at lower analyte concentrations. Careful attention to cleaning and handling procedures is required during all steps of sampling and analysis to minimize this source of error.

6.5 The general approaches listed below should be followed to minimize interferences.

6.5.1 Polar compounds, including certain pesticides (e.g., organophosphorus and carbamate classes) can be removed by column chromatography on alumina. Alumina clean-up will permit analysis of most organochlorine pesticides and PCBs (4).

6.5.2 PCBs may be separated from other organochlorine pesticides by column chromatography on silicic acid (5,6).

6.5.3 Many pesticides can be fractionated into groups by column chromatography on Florisil (6).

7. Equipment and Materials

7.1 Materials for Sample Collection

7.1.1 Continuous-Flow Sampling Pump (see Figure 1). The pump should provide a constant air flow ($\leq \pm 5\%$), be quiet and unobtrusive, with a flow rate of 1 to 5 L/min. Sources of equipment are Supelco, Supelco Park, Bellefonte, PA; SKC, 334 Valley View Road, Eighty Four, PA and other manufacturers.

7.1.2 Sampling Cartridge (see Figure 2). Constructed from a 20-mm (I.D.) x 10-cm borosilicate glass tube drawn down to a 7-mm (O.D.) open connection for attachment to the pump by way of flexible tubing (see Figure 1).

7.1.3 Sorbent, Polyurethane Foam (PUF). Cut into a cylinder, 22-mm I.D. and 7.6-cm long, fitted under slight compression inside the cartridge. The PUF should be of the polyether type, (density of 0.0225 g/cm^3). This is the type of foam used for furniture upholstery, pillows, and mattresses. The PUF cylinders (plugs) should be slightly larger in diameter than the internal diameter of the cartridge. The PUF sorbent may be cut by one of the following means:

- With a high-speed cutting tool, such as a motorized cork borer. Distilled water should be used to lubricate the cutting tool.
- With a hot wire cutter. Care should be exercised to prevent thermal degradation of the foam.
- With scissors, while plugs are compressed between the 22-mm circular templates.

Alternatively, pre-extracted PUF plugs and glass cartridges may be obtained commercially.

7.1.4 Particle Filter. The collection efficiency of PUF for small-diameter (0.1 to 1 μm) airborne particles is only about 20% (7). However, most pesticides and PCBs exist in air under steady-state conditions primarily as vapors (8). Most particulate-associated pesticides or PCBs, if any, will also tend to be vaporized from filters after collection (9). Collocated sampling with and without a quartz-fiber pre-filter has yielded indistinguishable results for a broad spectrum of pesticides and PCBs found in indoor air (10).

7.1.4.1 An open-face filter may be attached to the sampling cartridge by means of a union for 1-in. (25.4-mm) tubing.

7.1.4.2 A 32-mm diameter quartz microfiber filter (e.g., Palifelex® type 2500 QAT-UP) is placed in the open end of the union and supported by means of a screen or perforated metal plate [e.g., a 304-stainless steel disk, 0.0312-in. (0.8-mm) thick with 1/16-in. (1.6-mm) diameter round perforations at 132 holes per in.^{2} (20 holes/cm^2), 41% open area.]. A 32-mm Viton® O-ring is placed between the filter and outer nut to effect a seal (see Figure 3). This filter holder is available from Supelco Park, Bellefonte, PA; SKC, 334 Forty Eight, PA; and other manufacturers.

7.1.5 Size-Selective Impactor Inlet. A size-selective impactor inlet with an average particle-size cut-point of 2.5 μm or 10 μm mean diameter at a sampling rate of 4 L/min may be used to exclude nonrespirable airborne particulate matter (11). This inlet, particle filter support, sampling cartridge holders are available commercially from Supelco, Supelco Park, Bellefonte, PA; SKC, 334 Forty Eight, PA and University Research Glassware (URG), Chapel Hill, NC.

7.1.6 Tenax-TA. 60/80 mesh, 2,6-diphenylphenylene oxide polymer. Commercially available from Supelco, Supelco Park, Bellefonte, PA and SKC, 334 Forty Eight, PA.

7.2 Equipment for Analysis

7.2.1 Gas Chromatograph (GC). The GC system should be equipped with appropriate detector(s) and either an isothermally controlled or temperature programmed heating oven. Improved detection limits may be obtained with a GC equipped with a cool on-column or splitless injector.

7.2.2 Gas Chromatographic Column. As an example, a 0.32 mm (I.D.) x 30 m DB-5, DB-17, DB-608, and DB-1701 are available. Other columns may also provide acceptable results.

7.2.3 HPLC Column. As an example, a 4.6-mm x 25-cm Zorbax SIL or μBondpak C-18. Other columns may also provide acceptable results.

7.2.4 Microsyringes. 5 μL volume or other appropriate sizes.

7.3 Reagents and Other Materials

7.3.1 Round Bottom Flasks. 500 mL, ₸ 24/40 joints, best source.
7.3.2 Capacity Soxhlet Extractors. 300 mL, with reflux condensers, best source.
7.3.3 Kuderna-Danish Concentrator. 500 mL, with Snyder columns, best source.
7.3.4 Graduated Concentrator Tubes. 10 mL, with 19/22 stoppers, best source.
7.3.5 Graduated Concentrator Tubes. 1 mL, with 14/20 stoppers, best source.
7.3.6 TFE Fluorocarbon Tape. 1/2 in., best source.
7.3.7 Filter Tubes. Size 40 mm (I.D.) x 80 mm.
7.3.8 Serum Vials. 1 mL and 5 mL, fitted with caps lined with TFE fluorocarbon.
7.3.9 Pasteur Pipettes. 9 in., best source.
7.3.10 Glass Wool. Fired at 500°C, best source.
7.3.11 Boiling Chips. Fired at 500°C, best source..
7.3.12 Forceps. Stainless steel, 12 in., best source.
7.3.13 Gloves. Latex or precleaned (5% ether/hexane Soxhlet extracted) cotton.

7.3.14 Steam Bath.
7.3.15 Heating Mantles. 500 mL.
7.3.16 Analytical Evaporator. Nitrogen blow-down.
7.3.17 Acetone. Pesticide quality.
7.3.18 n-Hexane. Pesticide quality.
7.3.19 Diethyl Ether. Preserved with 2% ethanol.
7.3.20 Sodium Sulfate. Anhydrous analytical grade.
7.3.21 Alumina. Activity Grade IV, 100/200 mesh.
7.3.22 Glass Chromatographic Column. 2-mm I.D. x 15-cm long.
7.3.23 Soxhlet Extraction System. Including Soxhlet extractors (500 and 300 mL), variable voltage transformers, and cooling water source.
7.3.24 Vacuum Oven. Connected to water aspirator.
7.3.25 Die.
7.3.26 Ice Chest.
7.3.27 Silicic Acid. Pesticide grade.
7.3.28 Octachloronaphthalene (OCN). Research grade.
7.3.29 Florisil. Pesticide grade.

8. Assembly and Calibration of Sampling System

8.1 Description of Sampling Apparatus

8.1.1 A typical sampling arrangement utilizing a personal air pump is shown in Figure 1. This method is designed to use air sampling pumps capable of pulling air through the sampling cartridge at flow rates of 1 to 5 L/min. The method writeup presents the use of this device.
8.1.2 The sampling cartridge (see Figure 2) consists of a glass sampling cartridge in which the PUF plug or PUF/Tenax® TA "sandwich" is retained.

8.2 Calibration of Sampling System

8.2.1 Air flow through the sampling system is calibrated by the assembly shown in Figure 4. All air sampler must be calibrated in the laboratory before and after each sample collection period, using the procedure described below.
8.2.2 For accurate calibration, attach the sampling cartridge in-line during calibration. Vinyl bubble tubing or other means (e.g., rubber stopper or glass joint) may be used to connect the large end of the cartridge to the calibration system. Refer to ASTM Practice D3686 or D4185, for procedures to calibrate small volume air pumps.

9. Preparation of PUF Sampling Cartridges

9.1 The PUF adsorbent is white and yellows upon exposure to light. The "yellowing" of PUF will not affect its ability to collected pesticides or PCBs.
9.2 For initial cleanup and quality assurance purposes, the PUF plug is placed in a Soxhlet extractor and extracted with acetone for 14 to 24 hours at 4 to 6 cycles per hour.

[Note: If commercially pre-extracted PUF plugs are used, extraction with acetone is not required.]

Follow with a 16-hour Soxhlet extraction with 5% diethyl ether in n-hexane. When cartridges are reused, 5% diethyl ether in n-hexane can be used as the cleanup solvent.

9.3 Place the extracted PUF in a vacuum oven connected to a water aspirator and dry at room temperature for 2 to 4 hours (until no solvent odor is detected). Alternatively, they may be dried at room temperature in an air-tight container with circulating nitrogen (zero grade). Place the clean PUF plug into a labeled glass sampling cartridges using gloves and forceps. Wrap the cartridges with hexane-rinsed aluminum foil and placed in jars fitted with TFE fluorocarbon-lined caps. The foil wrapping may also be marked for identification using a blunt probe.

9.4 Granular sorbents may be combined with PUF to extend the range of use to compounds with saturation vapor pressures greater than 10^{-4} kPa (6). A useful combination trap can be assembled by "sandwiching" 0.6 g of Tenax-TA between two 22-mm I.D. x 3.8-cm pre-cleaned PUF plugs, as shown in Figure 2, Cartridge b. The Tenax-TA should be pre-extracted as described in Section 9.2. This trap may be extracted, vacuum dried, and removed without unloading it.

9.5 Analyze at least one assembled cartridge from each batch as a laboratory blank before the batch is acceptable. A blank level of <10 ng/plug for single component compounds is considered to be acceptable. For multiple component mixtures (e.g., PCBs), the blank level should be <100 ng/plug.

9.6 After cleaning, cartridges are considered clean up to 30 days when stored in sealed containers. Certified clean cartridges do not need to be chilled when shipping to the field.

10. Sampling

[Note: After the sampling system has been assembled and calibrated as per Section 8, it can be used to collect air samples as described below. The prepared sample cartridges should be used within 30 days of certification and should be handled only with latex or precleaned cotton gloves.]

10.1 Carefully remove the clean sample cartridge from the aluminum foil wrapping (the foil is returned to jars for later use) and attached to the pump with flexible tubing. The sampling assembly is positioned with the intake downward or in horizontal position. Locate the sampler in an unobstructed area at least 30 meters from any obstacle to air flow. The PUF or PUF/XAD-2 cartridge intake is positioned 1 to 2 m above ground level. Cartridge height above ground is recorded on the Compendium Method TO-10A field test data sheet (FTDS), as illustrated in Figure 5.

10.2 After the PUF cartridge is correctly inserted and positioned, the power switch is turned on and the sampling begins. The elapsed time meter is activated and the start time is recorded. The pumps are checked during the sampling process and any abnormal conditions discovered are recorded on the FTDS. Ambient temperatures and barometric pressures are measured and recorded periodically during the sampling procedure on the FTDS.

10.3 At the end of the desired sampling period, the power is turned off, the PUF cartridge removed from the sampler and wrapped with the original aluminum foil and placed in a sealed, labeled container for transport, under blue ice (<4°C), back to the laboratory. At least one field blank is returned to the laboratory with each group of

samples. A field blank is treated exactly like a sample except that no air is drawn through the cartridge. Samples are stored at <4°C or below until analyzed in the laboratory. Extraction must occur within 7 days of sampling and analysis within 40 days of extraction. Refer to ASTM D4861-94 (1), Appendix X3 for storage stability for various common pesticides and other compounds on PUF or PUF/Tenax TA sandwich.

11. Sample Extraction Procedure

[Note: Sample extraction should be performed under a properly ventilated hood.]

11.1 Sample Extraction

11.1.1 All samples should be extracted within 1 week after collection. All samples should be stored at <4°C until extracted.

11.1.2 All glassware should be washed with a suitable detergent; rinsed with deionized water, acetone, and hexane; rinsed again with deionized water; and fired in an oven (500°C).

11.1.3 Prepare a spiking solution for determination of extraction efficiency. The spiking solution should contain one or more surrogate compounds that have chemical structures and properties similar to those of the analytes of interest. Octachloronaphthalene (OCN) and dibutylchlorendate have been used as surrogates for determination of organochlorine pesticides by GC with an ECD. Tetrachloro-m-xylene and decachlorobiphenyl can also be used together to insure recovery of early and late eluting compounds. For organophosphate pesticides, tributylphosphate or triphenylphosphate may be employed as surrogates. The surrogate solution should be prepared so that addition of 100 μL into the PUF plug results in an extract containing the surrogate compound at the high end of the instrument's calibration range. As an example, the spiking solution for OCN is prepared by dissolving 10 mg of OCN in 10 mL of 10% acetone in n-hexane, followed by serial dilution n-hexane to achieve a final spiking solution of OCN of 1 μg/mL.

[Note: Use the recoveries of the surrogate compounds to monitor for unusual matrix effects and gross sample processing errors. Evaluate surrogate recovery for acceptance by determining whether the measured concentration falls within the acceptance limits of 60-120 percent.]

11.1.4 The extracting solution (5% diethyl ether/hexane) is prepared by mixing 1900 mL of freshly opened hexane and 100 mL of freshly opened diethyl ether (preserved with ethanol) to a flask.

11.1.5 All clean glassware, forceps, and other equipment to be used should be rinsed with 5% diethyl ether/hexane and placed on rinsed (5% diethyl ether/hexane) aluminum foil until use. The condensing towers should also be rinsed with 5% diethyl ether/hexane. Then add 300 mL or 5% diethyl ether/hexane to the 500 mL round bottom boiling flask and add up to three boiling granules.

11.1.6 Using precleaned (i.e., 5% diethyl ether/hexane Soxhlet extracted) cotton gloves, the glass PUF cartridges are removed from the sealed container, the PUF removed from the glass container and is placed into the 300 mL Soxhlet extractor using prerinsed forceps.

[Note: If "sandwich" trap is used, carefully clean outside walls of cartridge with hexane-soaked cotton swabs or laboratory tissues (discard) and place cartridge into extractor with intake (large end) downward.]

11.1.7 Before extraction begins, add 100 µL of the OCN solution directly to the top of the PUF plug.

[Note: Incorporating a known concentration of the solution onto the sample provides a quality assurance check to determine recovery efficiency of the extraction and analytical processes.]

11.1.8 Connect the Soxhlet extractor to the 500 mL boiling flask and condenser. Wet the glass joints with 5% diethyl ether/hexane to ensure a tight seal between the fittings. If necessary, the PUF plug can be adjusted using forceps to wedge it midway along the length of the siphon. The above procedure should be followed for all samples, with the inclusion of a blank control sample.

11.1.9 The water flow to the condenser towers of the Soxhlet extraction assembly should be checked and the heating unit turned on. As the samples boil, the Soxhlet extractors should be inspected to ensure that they are filling and siphoning properly (4 to 6 cycles/hour). Samples should cycle for a minimum of 16 hours.

11.1.10 At the end of the extracting process (minimum of 16 hours), the heating unit is turned off and the sample cooled to room temperature.

11.1.11 The extracts are then concentrated to 5 mL using a Kuderna-Danish (K-D) apparatus. The K-D is set up, assembled with concentrator tubes, and rinsed. The lower end of the filter tube is packed with glass wool and filled with sodium sulfate to a depth of 40 mm. The filter tube is then placed in the neck of the K-D. The Soxhlet extractors and boiling flasks are carefully removed from the condenser towers and the remaining solvent is drained into each boiling flask. Sample extract is carefully poured through the filter tube into the K-D. Each boiling flask is rinsed three times by swirling hexane along the sides. Once the sample has drained, the filter tube is rinsed down with hexane. Each Synder column is attached to the K-D and rinsed to wet the joint for a tight seal. The complete K-D apparatus is placed on a steam bath and the sample is evaporated to approximately 5 mL.

[Note: Do not allow samples to evaporate to dryness.]

Remove sample from the steam bath, rinse Synder column with minimum of hexane, and allow to cool. Adjust sample volume to 10 mL in a concentrator tube, close with glass stopper and seal with TFE fluorocarbon tape. Alternatively, the sample may be quantitatively transferred (with concentrator tube rinsing) to prescored vials and brought up to final volume. Concentrated extracts are stored at <4 °C until analyzed. Analysis should occur no later than 40 days after sample extraction.

11.2 Sample Cleanup

11.2.1 If polar compounds (from example, organophosphorus and carbamate classes) that interfere with GC/ECD analysis are present, use column chromatographic cleanup or alumina. The sample cleanup will permit the analysis of most organochlorine pesticides or PCBs.

11.2.2 Before cleanup, the sample extract is carefully reduced to 1 mL using a gentle stream of clean nitrogen.

11.2.3 A glass chromatographic column (2-mm I.D. x 15-cm long) is packed with alumina, activity grade IV, and rinsed with approximately 20 mL of n-hexane. The concentrated sample extract is placed on the column and eluted with 10 mL of n-hexane at a rate of 0.5 mL/minute. The eluate volume is adjusted to exactly 10 mL and analyzed as per Section 12.

11.2.4 If both PCBs and organochlorine pesticides are sought, alternate cleanup procedures (5,6) may be required (i.e., silicic acid).

11.2.5 Finally, class separation and improved specificity can be achieved by column clean-up and separation on Florisil (6).

12. Analytical Procedure

12.1 Analysis of Organochlorine Pesticides by Capillary Gas Chromatography with Electron Capture Detector (GC/ECD)

[Note: Organochlorine pesticides, PCBs and many nonchlorinated pesticides are responsive to electron capture detection (see Table 1). Most of these compounds can be analyzed at concentration of 1 to 50 ng/mL by GC/ECD. The following procedure is appropriate. Analytical methods that have been used to determine pesticides and PCBs collected from air by this procedure have been published (12).]

12.1.1 Select GC column (e.g., 0.3-mm by 30-m DB-5 column) and appropriate GC conditions to separate the target analytes. Typical operating parameters for this column with splitless injection are: Carrier gas-chromatography grade helium at a flow rate of 1 to 2 mL/min and a column head pressure of 7 to 9 psi (48 to 60 kPa); injector temperature of 250°C; detector temperature of 350°C; initial oven temperature of 50°C held for 2.0 min., ramped at 15°C/min to 150°C for 8 min, ramped at 10°C/min to 295°C then held for 5 min; purge time of 1.0 min. A typical injection volume is 2 to 3 μL.

12.1.2 Remove sample extract from the refrigerator and allow to warm to room temperature.

12.1.3 Prepare standard solution from reference materials of known purity. Analytically pure standards of organochlorine pesticides and PCBs are available from several commercial sources.

12.1.4 Use the standard solutions of the various compounds of interest to determine relative retention times (RRTs) to an internal standard such as p,p'-DDE, aldrin or octachloronaphthalene. Use 1 to 3-μL injections or other appropriate volumes.

12.1.5 Determine detector linearity by injecting standard solutions of three different concentrations (amounts) that bracket the range of analyses. The calibration is considered linear if the relative standard deviation (RSD) of the response factors for the three standards is 20 percent or less.

12.1.6 Calibrate the system with a minimum of three levels of calibration standards in the linear range. The low standard should be near the analytical method detection limit. The calibration is considered linear if the relative standard deviation (RSD) of the response factors for the three standards is 20 percent or less. The initial calibration should be verified by the analysis of a standard from an independent source. Recovery of 85 to 115 percent is acceptable. The initial calibration curve should be verified at the begining of each day and after every ten samples by the analysis of the mid point standard; an RPD of 15% or less is acceptable for continuing use of the initial calibration curve.

12.1.7 Inject 1 to 3 μL of the sample extract. Record volume injected to the nearest 0.05 μL.

12.1.8 A typical ECD response for a mixture of single component pesticides using a capillary column is illustrated in Figure 6. If the response (peak height or area) exceeds the calibration range, dilute the extract and reanalyze.

12.1.9 Quantify PCB mixtures by comparison of the total heights or areas of GC peaks (minimum of 5) with the corresponding peaks in the best-matching standard. Use Aroclor 1242 for early-eluting PCBs and either Aroclor 1254 or Aroclor 1260 as appropriate for late-eluting PCBs.

12.1.10 If both PCBs and organochlorine pesticides are present in the same sample, use column chromatographic separation on silicic acid (5,6) prior to GC analysis.

12.1.11 If polar compounds are present that interfere with GC/ECD analysis, use column chromatographic cleanup or alumina, activity grade IV, in accordance with Section 11.2.

12.1.12 For confirmation use a second GC column such as DB-608. All GC procedures except GC/MS require second column confirmation.

12.1.13 For improved resolution use a capillary column such as an 0.25-mm I.D. x 30-m DB-5 with 0.25 μm film thickness. The following conditions are appropriate.

- Helium carrier gas at 1 mL/min.
- Column temperature program, 90°C (4 min)/16°C/min to 154°C/4°C/min to 270°C.
- Detector, ^{63}Ni ECD at 350°C.
- Make up gas, nitrogen, or 5% methane/95% argon at 60 mL/min.
- Splitless injection, 2 μL maximum.
- Injector temperature, 220°C.

12.1.14 Class separation and improved specificity can be achieved by column chromatographic separation on Florisil (6).

12.2 Analysis of Organophosphorus Pesticides by Capillary Gas Chromatography with Flame Photometric or Nitrogen-Phosphorus Detectors (GC/FPD/NPD)

[Note: Organophosphorus pesticides are responsive to flame photometric and nitrogen-phosphorus (alkali flame ionization) detection. Most of these compounds can be analyzed at concentrations of 50 to 500 ng/mL using either of these detectors.]

12.2.1 Procedures given in Section 12.1.1 through 12.1.9 and Section 12.1.13 through 12.1.14 apply, except for the selection of surrogates.

12.2.2 Use tributylphosphate, triphenylphosphate, or other suitable compound(s) as surrogates to verify extraction efficiency and to determine RRTs.

12.3 Analysis of Carbamate and Urea Pesticides by Capillary Gas Chromatography with Nitrogen-Phosphorus Detector

12.3.1 Trazine, carbamate, and urea pesticides may be determined by capillary GC (DB-5, DB-17, or DB-1701 stationary phase) using nitrogen-phosphorus detection or MS-SIM with detection limits in the 0.05 to 0.2 μL/mL range. Procedures given in Section 12.1.1 through 12.1.9 and Section 12.1.13 through 12.1.14 apply, except for the selection of surrogates, detector, and make up gas.

12.3.2 Thermal degradation may be minimized by reducing the injector temperature to 200°C. HPLC may also be used, but detection limits will be higher (1 to 5 μg/mL).

12.3.3 N-methyl carbamates may be determined using reverse-phase high performance liquid chromatography (HPLC) (C-18) (Section 12.4) and post-column derivatization with o-phthaldehyde and fluorescence detection (EPA Method 531). Detection limits of 0.01 to 0.1 μg/mL can be achieved.

12.4 Analysis of Carbamate, Urea, Pyrethroid, and Phenolic Pesticides by High Performance Liquid Chromatography (HPLC)

[Note: Many carbamate pesticides, urea pesticides, pyrethrins, phenols, and other polar pesticides may be analyzed by high HPLC with fixed or variable wavelength UV detection. Either reversed-phase or normal phase chromatography may be used. Detection limits are 0.2 to 10 μg/mL of extract.]

12.4.1 Select HPLC column (i.e., Zorbax-SIL, 46-mm I.D. x 25-cm, or μ-Bondapak C18, 3.9-mm x 30-cm, or equivalent).

12.4.2 Select solvent system (i.e., mixtures of methanol or acetonitrile with water or mixtures of heptane or hexane with isopropanol).

12.4.3 Follow analytical procedures given in Sections 12.1.2 through 12.1.9.

12.4.4 If interferences are present, adjust the HPLC solvent system composition or use column chromatographic clean-up with silica gel, alumina, or Florisil (6).

12.4.5 An electrochemical detector may be used to improve sensitivity for some ureas, carbamates, and phenolics. Much more care is required in using this detector, particularly in removing dissolved oxygen from the mobile phase and sample extracts.

12.4.6 Chlorophenol (di- through penta-) may be analyzed by GC/ECD or GC/MS after derivatization with pentafluorobenzylbromide (EPA Method 604).

12.4.7 Chlorinated phenoxyacetic acid herbicides and pentachlorophenol can be analyzed by GC/ECD or GC/MS after derivatization with diazomethane (EPA Method 515). DB-5 and DB-1701 columns (0.25-mm I.D. x 30-m) at 60 to 300°C/4°C per min have been found to perform well.

12.5 Analysis of Pesticides and PCBs by Gas Chromatography with Mass Spectrometry Detection (GC/MS)

[Note: A mass spectrometer operating in the selected ion monitoring mode is useful for confirmation and identification of pesticides.]

12.5.1 A mass spectrometer operating in the select ion monitoring (SIM) mode can be used as a sensitive detector for multi-residue determination of a wide variety of pesticides. Mass spectrometers are now available that provide detection limits comparable to nitrogen-phosphorus and electron capture detectors.

12.5.2 Most of the pesticides shown in Table 1 have been successfully determined by GC/MS/SIM. Typical GC operating parameters are as described in Section 12.1.1.

12.5.3 The mass spectrometer is typically operated using positive ion electron impact ionization (70 eV). Other instrumental parameters are instrument specific.

12.5.4 p-Terphenyl-d_{14} is commonly used as a surrogate for GC/MS analysis.

12.5.5 Quantification is typically performed using an internal standard method. 1,4-Dichlorobenzene, naphthalene-d_8, acenaphthene-d_{10}, phenanthrene-d_{10}, chrysene-d_{12} and perylene-d_{12} are commonly used as internal standards. Procedures given in Section 12.1.1 through 12.1.9 and Section 12.1.13 through 12.1.14 apply, except for the selection of surrogates, detector, and make up gas.

12.5.6 See ASTM Practice D 3687 for injection technique, determination of relative retention times, and other procedures pertinent to GC and HPLC analyses.

12.6 Sample Concentration

12.6.1 If concentrations are too low to detect by the analytical procedure of choice, the extract may be concentrated to 1 mL or 0.5 mL by carefully controlled evaporation under an inert atmosphere. The following procedure is appropriate.

12.6.2 Place K-D concentrator tube in a water bath and analytical evaporator (nitrogen blow-down) apparatus. The water bath temperature should be from 25°C to 50°C.

12.6.3 Adjust nitrogen flow through hypodermic needle to provide a gentle stream.

12.6.4 Carefully lower hypodermic needle into the concentrator tube to a distance of about 1 cm above the liquid level.

12.6.5 Continue to adjust needle placement as liquid level decreases.

12.6.6 Reduce volume to slightly below desired level.

12.6.7 Adjust to final volume by carefully rinsing needle tip and concentrator tube well with solvent (usually n-hexane).

13. Calculations

13.1 Determination of Concentration

13.1.1 The concentration of the analyte in the extract solution can be taken from a standard curve where peak height or area is plotted linearly against concentration in nanograms per milliliter (ng/mL). If the detector response is known to be linear, a single point is used as a calculation constant.

13.1.2 From the standard curve, determine the nanograms of analyte standard equivalent to the peak height or area for a particular compound.

13.1.3 Ascertain whether the field blank is contaminated. Blank levels should not exceed 10 ng/sample for organochlorine pesticides or 100 ng/sample for PCBs and other pesticides. If the blank has been contaminated, the sampling series must be held suspect.

13.1.4 Quantity of the compound in the sample (A) is calculated using the following equation:

$$A = 1000 \left(\frac{A_s \times V_e}{V_i} \right)$$

where:

A = total amount of analyte in the sample, ng.

A_s = calculated amount of material injected onto the chromatograph based on calibration curve for injected standards, ng.

V_e = final volume of extract, mL.

V_i = volume of extract injected, μL.

1000 = factor for converting microliters to milliliters.

13.1.5 The extraction efficiency (EE) is determined from the recovery of surrogate spike as follows:

$$EE(\%) = \left| \frac{S}{S_a} \right| [100]$$

where:

EE = extraction efficiency, %.

S = amount of spike recovered, ng.

S_a = amount of spike added to plug, ng.

The extraction efficiency (surrogate recovery) must fall between 60-120% to be acceptable.

13.1.6 The total volume of air sampled under ambient conditions is determined using the following equation:

$$V_a = \frac{\sum_{i=1}^{n} (T_i \times F_i)}{1000 \text{ L/m}^3}$$

where:

V_a = total volume of air sampled, m^3.
T_i = length of sampling segment between flow checks, min.
F_i = average flow during sampling segment, L/min.

13.1.7 The air volume is corrected to EPA standard temperature (25 °C) and standard pressure (760 mm Hg) as follows:

$$V_s = V_a \left(\frac{P_b - P_w}{760 \text{ mm Hg}} \right) \left(\frac{298K}{t_A} \right)$$

where:

V_s = volume of air at standard conditions (25 °C and 760 mm Hg), std. m^3.
V_a = total volume of air sampled, m^3.
P_b = average ambient barometric pressure, mm Hg.
P_w = vapor pressure of water at calibration temperature, mm Hg.
t_A = average ambient temperature, °C + 273.

13.1.8 If the proper criteria for a sample have been met, concentration of the compound in a standard cubic meter of air sampled is calculated as follows:

$$C_a(\text{ng/std. m}^3) = \left[\frac{(A)}{(V_s)} \right] \left[\frac{(100)}{(SE(\%))} \right]$$

where:

SE = sampling efficiency as determined by the procedure outlined in Section 14.

If it is desired to convert the air concentration value to parts per trillion (ppt) in dry air at standard temperature and pressure (STP), the following conversion is used:

$$\text{ppt} = 0.844 \, (C_a)$$

The air concentration can be converted to parts per trillion (v/v) in air at STP as follows:

$$\text{pptv} = \left[\frac{(24.45) \, (C_a)}{(MW)} \right]$$

where:

MW = molecular weight of the compound of interest, g/g-mole.

13.1.9 If quantification is performed using an internal standard, a relative response factor (RRF) is calculated by the equation:

$$RRF = \left[\frac{(I_s)(C_{is})}{(I_{is})(C_s)}\right]$$

where:

I_s = integrated area of the target analyte peak, counts.
I_{is} = integrated area of the internal standard peak, counts.
C_{is} = concentration of the internal standard, ng/μL.
C_s = concentration of the analyte, ng/µL.

13.1.10 The concentration of the analyte (C_a) in the sample is then calculated as follows:

$$C_a = \frac{(I_s)(C_{is})}{(RRF)(I_{is})}$$

where:

C_a = concentration of analyte, ng/m^3
I_s = integrated area of the target analyte peak, counts.
RRF = relative response factor (see Section 13.1.10).

14. Sampling and Retention Efficiencies

14.1 General

14.1.1 Before using Compendium Method TO-10A, the user should determine the sampling efficiency for the compound of interest. The sampling efficiencies shown in Tables 2, 3, 4, and 5 were determined for approximately 1 m^3 of air at about 25 °C, sampled at 3.8 L/min. The SE values in these tables may be used for similar sampling conditions; for other compounds or conditions, SE values must be determined.

14.1.2 Sampling efficiencies for the pesticides shown in Table 6 are for a flowrate of 3.8 L/min and at 25 °C. For compounds not listed, longer sampling times, different flow rates, or other air temperatures, the following procedure may be used to determine sampling efficiencies.

14.2 Determining SE

14.2.1 SE is determined by a modified impinger assembly attached to the sampler pump, as illustrated in Figure 7. A clean PUF is placed in the pre-filter location and the inlet is attached to a nitrogen line.

[Note: Nitrogen should be used instead of air to prevent oxidation of the compounds under test. The oxidation would not necessarily reflect what may be encountered during actual sampling and may give misleading sampling efficiencies.]

Two PUF plugs (22-mm x 7.6-cm) are placed in the primary and secondary traps and are attached to the pump.

14.2.2 A standard solution of the compound of interest is prepared in a volatile solvent (i.e., hexane, pentane, or benzene). A small, accurately measured volume (i.e., 1 mL) of the standard solution is placed into the modified midget impinger. The sampler pump is set at the rate to be used in field application and then activated. Nitrogen is drawn through the assembly for a period of time equal to or exceeding that intended for field application. After the desired sampling test period, the PUF plugs are removed and analyzed separately as per Section 12.

14.2.3 The impinger is rinsed with hexane or another suitable solvent and quantitatively transferred to a volumetric flask or concentrator tube for analysis.

14.2.4 The sampling efficiency (SE) is determined using the following equation:

$$\% \text{ SE} = \frac{W_1}{W_0 - W_r} \times 100$$

where:

W_1 = amount of compound extracted from the primary trap, ng.

W_o = original amount of compound added to the impinger, ng.

W_r = residue left in the impinger at the end of the test, ng.

14.2.5 If material is found in the secondary trap, it is an indication that breakthrough has occurred. The addition of the amount found in the secondary trap, W_2, to W_1, will provide an indication for the overall sampling efficiency of a tandem-trap sampling system. The sum of W_1, W_2 (if any), and W_r must equal (approximately ±10%) W_o or the test is invalid.

14.2.6 If the compound of interest is not sufficiently volatile to vaporize at room temperature, the impinger may be heated in a water bath or other suitable heater to a maximum of 50°C to aid volatilization. If the compound of interest cannot be vaporized at 50°C without thermal degradation, dynamic retention efficiency (RE_d) may be used to estimate sampling efficiency. Dynamic retention efficiency is determined in the manner described in Section 14.2.7. Table 7 lists those organochlorine pesticides which dynamic retention efficiencies have been determined.

14.2.7 A pair of PUF plugs is spiked by slow, dropwise addition of the standard solution to one end of each plug. No more than 0.5 to 1 mL of solution should be used. Amounts added to each plug should be as nearly the same as possible. The plugs are allowed to dry for 2 hours in a clean, protected place (i.e., desiccator). One spiked plug is placed in the primary trap so that the spiked end is at the intake and one clean unspiked plug is placed in the secondary trap. The other spiked plug is wrapped in hexane-rinsed aluminum foil and stored in a clean place for the duration of the test (this is the static control plug, Section 14.2.8). Prefiltered nitrogen or ambient air is drawn through the assembly as per Section 14.2.2.

[Note: Impinger may be discarded.]

Each PUF plug (spiked and static control) is analyzed separately as per Section 12.

14.2.8 This dynamic retention efficiency (% RE_d) is calculated as follows:

$$\% \text{ RE}_d = \frac{W_1}{W_o} \times 100$$

where:

W_1 = amount of compound recovered from primary plug, ng.

W_o = amount of compound added to primary plug, ng.

If a residue, W_2, is found on the secondary plug, breakthrough has occurred. The sum of $W_1 + W_2$ must equal W_o, within 25% or the test is invalid. For most compounds tested by this procedure, % RE_d values are generally less than % SE values determined per Section 14.2. The purpose of the static RE_d determination is to establish any loss or gain of analyte unrelated to the flow of nitrogen or air through the PUF plug.

15. Performance Criteria and Quality Assurance

[Note: This section summarizes required quality assurance (QA) measures and provides guidance concerning performance criteria that should be achieved within each laboratory.]

15.1 Standard Operating Procedures (SOPs)

15.1.1 Users should generate SOPs describing the following activities accomplished in their laboratory: (1) assembly, calibration, and operation of the sampling system, with make and model of equipment used; (2) preparation, purification, storage, and handling of sampling cartridges; (3) assembly, calibration, and operation of the analytical system, with make and model of equipment used; and (4) all aspects of data recording and processing, including lists of computer hardware and software used.

15.1.2 SOPs should provide specific stepwise instructions and should be readily available to, and understood by, the laboratory personnel conducting the work.

15.2 Process, Field, and Solvent Blanks

15.2.1 One PUF cartridge from each batch of approximately twenty should be analyzed, without shipment to the field, for the compounds of interest to serve as a process blank.

15.2.2 During each sampling episode, at least one PUF cartridge should be shipped to the field and returned, without drawing air through the sampler, to serve as a field blank.

15.2.3 Before each sampling episode, one PUF plug from each batch of approximately twenty should be spiked with a known amount of the standard solution. The spiked plug will remain in a sealed container and will not be used during the sampling period. The spiked plug is extracted and analyzed with the other samples. This field spike acts as a quality assurance check to determine matrix spike recoveries and to indicate sample degradation.

15.2.4 During the analysis of each batch of samples, at least one solvent process blank (all steps conducted but no PUF cartridge included) should be carried through the procedure and analyzed.

15.2.5 All blank levels should not exceed 10 ng/sample for single components or 100 ng/sample for multiple component mixtures (i.e., for organochlorine pesticides and PCBs).

15.3 Sampling Efficiency and Spike Recovery

15.3.1 Before using the method for sample analysis, each laboratory must determine its sampling efficiency for the component of interest as per Section 14.

15.3.2 The PUF in the sampler is replaced with a hexane-extracted PUF. The PUF is spiked with a microgram level of compounds of interest by dropwise addition of hexane solutions of the compounds. The solvent is allowed to evaporate.

15.3.3 The sampling system is activated and set at the desired sampling flow rate. The sample flow is monitored for 24 hours.

15.3.4 The PUF cartridge is then removed and analyzed as per Section 12.

15.3.5 A second sampler, unspiked, is collected over the same time period to account for any background levels of components in the ambient air matrix.

15.3.6 In general, analytical recoveries and collection efficiencies of 75% are considered to be acceptable method performance.

15.3.7 Replicate (at least triplicate) determinations of collection efficiency should be made. Relative standard deviations for these replicate determinations of ±15% or less are considered acceptable performance.

15.3.8 Blind spiked samples should be included with sample sets periodically as a check on analytical performance.

15.4 Method Precision and Bias

15.4.1 Precision and bias in this type of analytical procedure are dependent upon the precision and bias of the analytical procedure for each compound of concern, and the precision and bias of the sampling process.

15.4.2 Several different parameters involved in both the sampling and analysis steps of this method collectively determine the precision and bias with which each compound is detected. As the volume of air sampled is increased, the sensitivity of detection increases proportionately within limits set by: (a) the retention efficiency for each specific component trapped on the polyurethane foam plug, and (b) the background interference associated with the analysis of each specific component at a given site sampled. The sensitivity of detection of samples recovered by extraction depends on: (a) the inherent response of the particular GC detector used in the determinative step, and (b) the extent to which the sample is concentrated for analysis. It is the responsibility of the analyst(s) performing the sampling and analysis steps to adjust parameters so that the required detection limits can be obtained.

15.4.3 The reproducibility of this method for most compounds for which it has been evaluated has been determined to range from ±5 to ±30% (measured as the relative standard deviation) when replicate sampling cartridges are used (N>5). Sample recoveries for individual compounds generally fall within the range of 90 to 110%, but recoveries ranging from 65 to 125% are considered acceptable. PUF alone may give lower recoveries for more volatile compounds (i.e., those with saturation vapor pressures $>10^{-3}$ mm Hg). In those cases, another sorbent or a combination of PUF and Tenax TA (see Figure 2) should be employed.

15.5 Method Safety

15.5.1 This procedure may involve hazardous materials, operations, and equipment. This method does not purport to address all of the safety problems associated with its use.

15.5.2 It is the user's responsibility to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to the implementation of this procedure. This should be part of the user's SOP manual.

16. References

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TABLE 1. COMPOUNDS FOR WHICH PROCEDURE HAS BEEN TESTED[1]

Compound	Recommended Analysis[2]	Compound	Recommended Analyses
Alachlor	GC/ECD	Heptachlor	GC/ECD
Aldrin	GC/ECD	Heptachlor epoxide	GC/ECD
Allethrin	HPLC/UV	Hexachlorobenzene	GC/ECD
Aroclor 1242	GC/ECD	Hexachlorocyclopentadiene[3,4]	GC/ECD
Aroclor 1254	GC/ECD	Lindane (γ-BHC)	GC/ECD
Aroclor 1260	GC/ECD	Linuron	HPLC/UV
Atrazine	GC/NPD	Malathion	GC/NPD or FPD
Bendiocarb	HPLC/UV	Methyl parathion	GC/NPD or FPD
BHC (α- and β-Hexachlorocyclohexanes)	GC/ECD	Methoxychlor	GC/FCD
Captan	GC/ECD	Metolachlor	GC/ECD
Carbaryl	HPLC/UV	Mexacarbate	GC/FCD
Carbofuran	HPLC/UV	Mirex	GC/ECD
Chlordane, technical	GC/ECD	Monuron	HPLC/UV
Chlorothalonil	GC/ECD	Trans-nonachlor	GC/ECD
Chlorotoluron	HPLC/UV	Oxychlordane	GC/ECD
Chlorpyritos	GC/ECD	Pentachlorobenzene	GC/ECD
2,4-D esters and salts	GC/ECD	Pentachlophenol	GC/ECD
Dacthal	GC/ECD	Permethrin (cis and trans)	HPLC/UV
ρ,ρ-'DDT	GC/ECD	o-Phenylphenol	HPLC/UV
ρ,ρ-'DDE	GC/ECD	Phorate	GC/NPD or FPD
Diazinon	GC/NPD or FPD	Propazine	GC/NPD
Dicloran	GC/ECD	Propoxur (Baygon)	HPLC/UV
Dieldrin	GC/ECD	Pyrethrin	HPLC/UV
Dichlorovos (DDVP)	GC/ECD	Resmethrin	HPLC/UV
Dicofol	GC/ECD	Ronnel	GC/ECD
Dicrotophos	HPLC/UV	Simazine	HPLC/UV
Diuron	HPLC/UV	Terbuthiuron	HPLC/UV
Ethyl parathion	GC/NPD or FPD	1,2,3,4-tetrachlorobenzene[3]	GC/ECD
Fenvalerate	HPLC/UV	1,2,3-trichlorobenzene[3]	GC/ECD
Fluometuron	HPLC/UV	2,3,5-trichlorophenol	GC/ECD
Folpet	GC/ECD	Trifluralin	GC/ECD

[1]The following recommendations are specific for that analyte for maximum sensitivity.
[2]GC = gas chromatography; ECD = electron capture detector, FPD = flame photometric detector; HPLC = high performance liquid chromatography; NPD = nitrogen-phosphorus detector; UV = ultraviolet absorption detector, (GC/MS (gas chromatography/mass spectrometry) may also be used).
[3]Using PUF/Tenax-TA "sandwich" trap.
[4]Compound is very unstable in solution.

TABLE 2. SAMPLING EFFICIENCIES FOR SOME ORGANOCHLORINE PESTICIDES

Compound	Quantity Introduced, μg^2	Air Volume, m^3	Sampling efficiency, %		
			mean	RSD	n
α-Hexachlorocyclohexane (α-BHC)	0.005	0.9	115	8	6
γ-Hexachlorocyclohexane (Lindane)	0.05-1.0	0.9	91.5	8	5
Chlordane, technical	0.2	0.9	84.0	11	8
p,p'-DDT	0.6, 1.2	0.9	97.5	21	12
p,p'-DDE	0.2, 0.4	0.9	102	11	12
Mirex	0.6, 1.2	0.9	85.9	22	7
2,4-D Esters:					
Isopropyl	0.5	3.6	92.0	5	12
Butyl	0.5	3.6	82.0	10	11
Isobutyl	0.5	3.6	79.0	20	12
Isoctyl	0.5	3.6	>80[2]	--	--

[1]Air volume = 0.9 m^3.
[2]Not vaporized. Value base on %RE = 81.0 (RSD = 10%, n = 6).

TABLE 3. SAMPLING EFFICIENCIES FOR ORGANOPHOSPHORUS PESTICIDES

Compound	Quantity Introduced, μg^2	Sampling efficiency, %		
		mean	RSD	n
Dichlorvos (DDVP)	0.2	72.0	13	2
Ronnel	0.2	106	8	12
Chlorpyrifos	0.2	108	9	12
Diazinon[1]	1.0	84.0	18	18
Methyl parathion[1]	0.6	80.0	19	18
Ethyl parathion[1]	0.3	75.9	15	18
Malathion[1]	0.3	100[3]	--	--

[1]Analyzed by gas chromatography with nitrogen phosphorus detector or flame photometric detector.
[2]Air volume = 0.9 m^3.
[3]Decomposed in generator; value based on %RE = 101 (RDS = 7, n = 4).

TABLE 4. SAMPLING EFFICIENCIES FOR SOME SEMI-VOLATILE ORGANOCHLORINE COMPOUNDS AND PCBs

Compound	Quantity Introduced, μg[1]	Sampling efficiency, %		
		mean	RSD	n
1,2,3-Trichlorobenzene	1.0	6.6[2]	22	8
1,2,3,4-Tetrachlorobenzene	1.0	62.3[2]	33	5
Pentachlorobenzene	1.0	94.0	12	5
Hexachlorobenzene	0.5, 1.0	94.5	8	5
Hexachlorocyclopentadiene	1.0	8.3[2]	12	5
2,4,5-Trichlorophenol	1.0	108	3	5
Pentachlorophenol	1.0	107	16	5
Aroclor 1242	0.1	96.0	15	6
Aroclor 1254	0.1	95.0	7	6
Aroclor 1260	0.1	109	5	11

[1]Air volume = 0.9 m^3.
[2]% SEs were 98, and 97% (n = 2), respectively, for these three compounds by the PUF/Tenax® TA "sandwich" trap.

TABLE 5. SAMPLING EFFICIENCIES FOR CARBAMATES, UREAS, TRIAZINES, AND PYRETHRINS

Compound	Spike Level, µg/plug	Static Recovery, %		n	Retention Efficiency, %		n	Sampling Efficiency, %		n
		mean	RSDP		mean	RSD		mean	RSD	
Carbamates:										
Propoxur	5	61.4	10	6	77.6	37	6	96.7	11	6
Carbofuran	15	55.3	12	6	64.2	46	6	87.2	14	6
Bendicarb	50	57.3	11	6	69.8	43	6	62.1	14	6
Mexacarbate	10	62.8	19	6	62.7	41	6	89.8	14	6
Carbaryl	100	56.6	14	6	63.6	53	6	0	13	6
Ureas:										
Monuron	19	87.0	6	6	91.2	6	5	0		
Diuron	20	84.1	8	6	90.0	2	5	0		
Linuron	20	86.7	8	6	92.5	4	5	0		
Terbuthiuron	18	85.0	8	6	88.8	8	5	0		
Fluometuron	20	91.4	10	6	101	3	5	0		
Chlortoluron	20	86.2	11	6	92.0	7	5	0		
Triazines:										
Simazine	10	103	6	5	101	9	6	0		
Atrazine	10	104	7	5	98.9	7	6	0		
Propazine	10	105	11	5	99.9	14	6	0		
Pyrethrins:										
Pyrethrin I	(9.7)	90.5	10	6	95.6	22	5	0		
Pyrethrin II	(6.1)	88.6	11	6	69.9	29	5	0		
Allethrin	25	69.2	9	5	58.3	12	6	0		
d-trans-Allethrin	25	76.8	9	6	74.4	9	5	0		
Dicrotophos	25	72.0	22	6	71.7	8	5	0		
Resmethrin	25	76.5	14	6	66.7	14	6	0		
Fenvalerate	25	87.9	3	6	57.2	20	3	0		

TABLE 6. EXTRACTION AND 24-H SAMPLING EFFICIENCIES FOR VARIOUS PESTICIDES AND RELATED COMPOUNDS

Compound	Extraction Efficiency[1], %		Sampling Eficiency[2], %, at					
			10 ng/m^3		100 ng/m^3		1,000 ng/m^3	
	mean	RSD	mean	RSD	mean	RSD	mean	RSD
Chlropyrifos	83.3	11.5	83.7	18.0	92.7	15.1	83.7	18.0
Pentachlorophenol	84.0	22.6	66.7	42.2	52.3	36.2	66.7	42.2
Chlordane	95.0	7.1	96.0	1.4	74.0	8.5	96.0	1.4
o-Phenylphenol	47.0	46.7	46.0	19.1	45.3	29.9	46.0	19.1
Lindane	96.0	6.9	91.7	11.6	93.0	2.6	91.7	11.6
DDVP	88.3	20.2	51.0	53.7	106.0	1.4	51.0	53.7
2,4-D Methyl Ester	--	--	75.3	6.8	58.0	23.6	75.3	6.8
Heptachlor	99.0	1.7	97.3	13.6	103.0	17.3	97.3	13.6
Aldrin	97.7	4.0	90.7	5.5	94.0	2.6	90.7	5.5
Dieldrin	95.0	7.0	82.7	7.6	85.0	11.5	82.7	7.6
Ronnel	80.3	19.5	74.7	12.1	60.7	15.5	74.7	12.2
Diazinon	72.0	21.8	63.7	18.9	41.3	26.6	63.7	19.9
trans-Nonachlor	97.7	4.0	96.7	4.2	101.7	15.3	96.7	4.2
Oxychlorodane	100.0	0.0	95.3	9.5	94.3	1.2	95.3	9.5
α-BHC	98.0	3.5	86.7	13.7	97.0	18.2	86.7	13.7
Bendiocarb	81.3	8.4	59.7	16.9	30.7	23.5	59.7	16.9
Chlorothalonil	90.3	8.4	76.7	6.1	70.3	6.5	76.7	6.1
Heptachlor Epoxide	100.0	0.0	95.3	5.5	97.7	14.2	95.3	5.5
Dacthal	--	--	87.0	9.5	95.3	22.2	87.0	9.5
Aroclor 1242	91.7	14.4	95.0	15.5	94.7	17.5	95.0	15.5

[1]Mean values for one spike at 550 ng/plug and two spikes at 5,500 ng/plug.
[2]Mean values for three determinations.

TABLE 7. EXTRACTION AND 24-H DYNAMIC RETENTION EFFICIENCIES FOR VARIOUS PESTICIDES AND RELATED COMPOUNDS

Compound	Extraction Efficiency[1], %		Sampling Eficiency[2], %, at					
			10 ng/m^3		100 ng/m^3		1,000 ng/m^3	
	mean	RSD	mean	RSD	mean	RSD	mean	RSD
Propoxur	77.5	71.4	92.0	--	91.7	22.8	101.0	18.4
Resmethrin	95.5	71.4	79.0	--	100.7	13.1	107.0	4.4
Dicofol	57.0	8.5	38.0	25.9	65.0	8.7	69.0	--
Captan	73.0	12.7	56.0	--	45.5	64.3	84.3	16.3
Carbaryl	74.0	82.0	102.0	--	61.0	--	113.0	6.1
Malathion	76.5	44.5	108.0	--	54.0	16.0	77.3	7.6
cis-Permethrin	88.7	10.3	101.0	28.5	85.0	26.9	89.0	11.3
trans-Permethrin	88.7	11.0	67.3	34.8	80.7	56.4	108.3	9.5
Methoxychlor	65.5	4.9	--	--	--	--	78.5	2.1
Atrazine	75.0	50.5	--	--	73.0	30.1	83.0	9.5
Folpet	86.7	11.7	--	--	78.0	--	93.0	--
Aroclor 1260	92.0	14.5	88.0	9.6	85.3	9.9	107.1	13.6

[1]Mean values for one spike at 550 ng/plug and two spikes at 5,500 ng/plug.
[2]Mean values for three determinations.

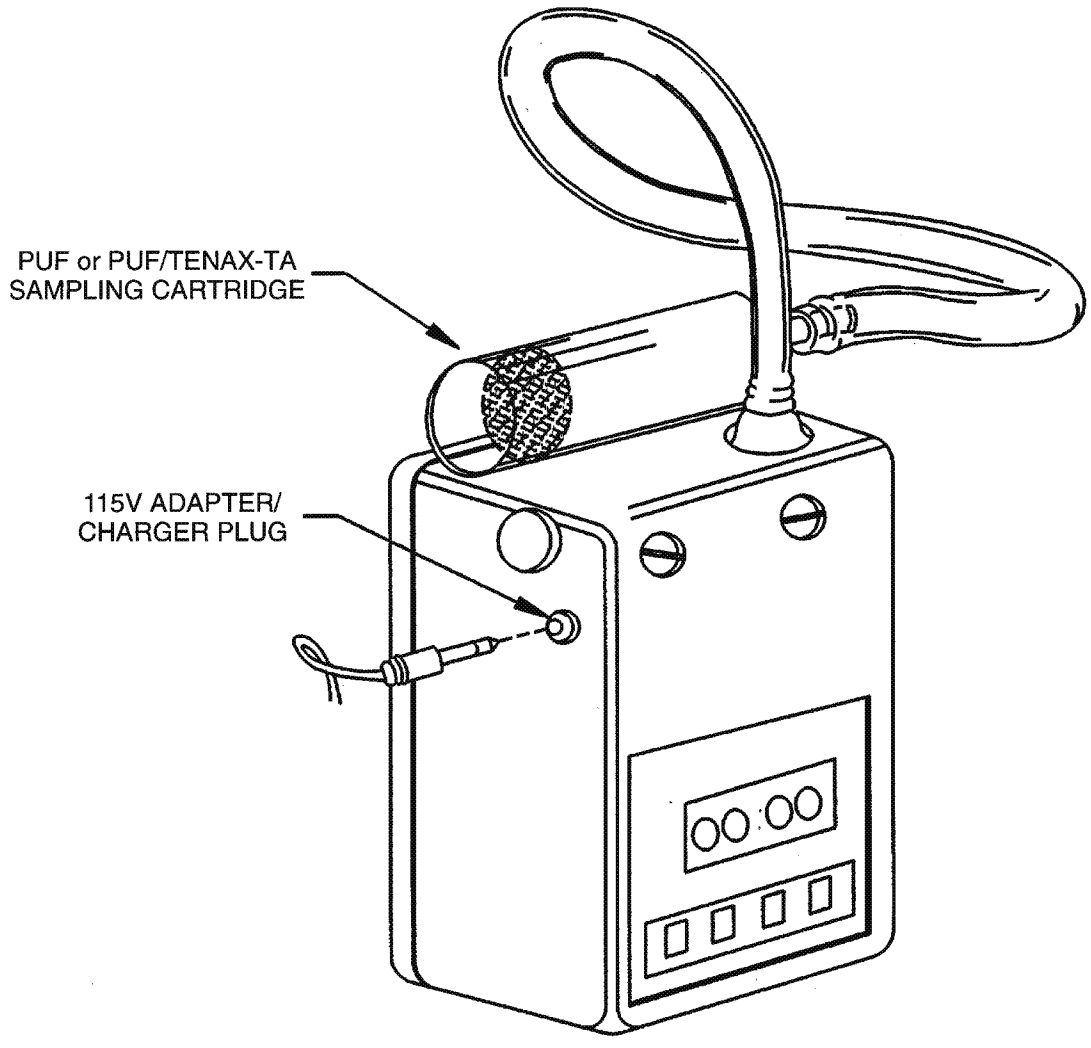

Figure 1. Low volume air sampler.

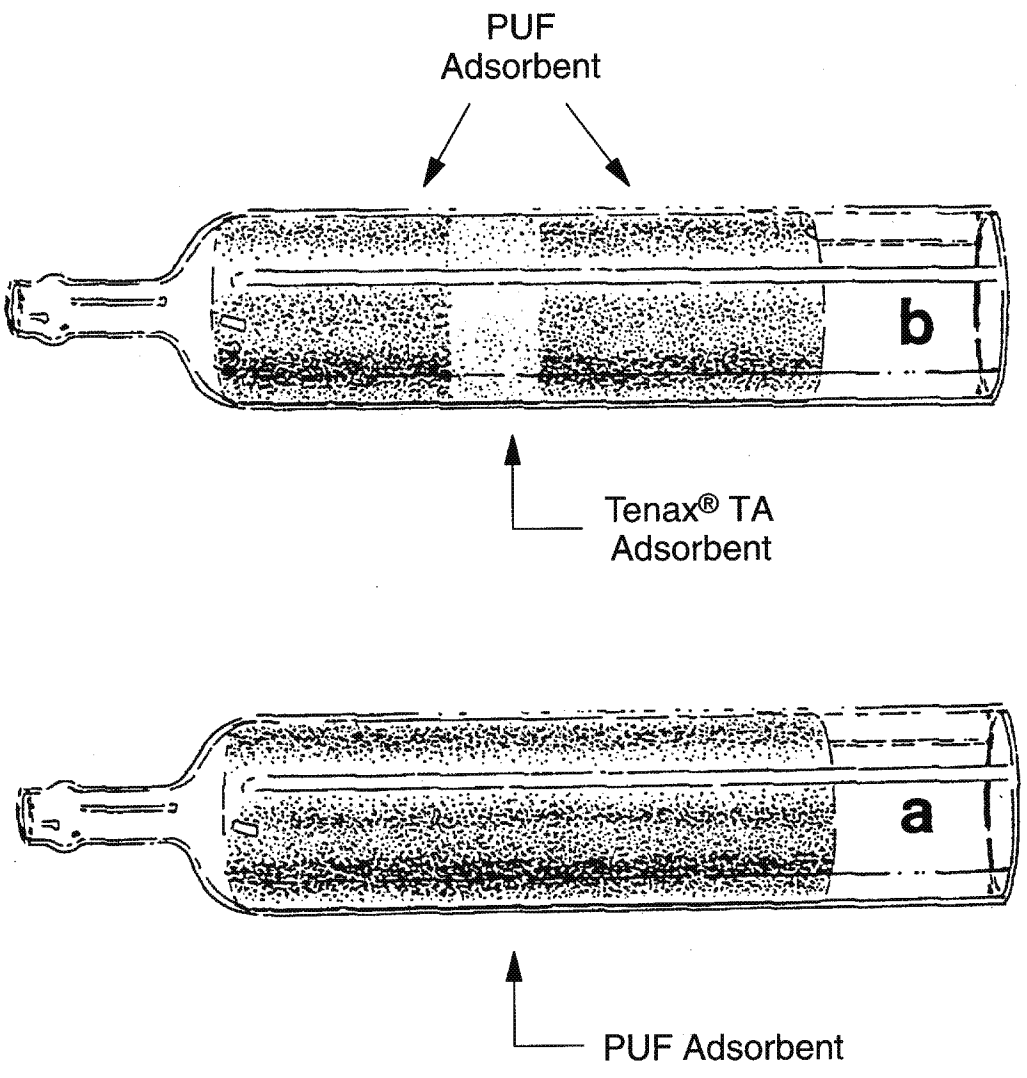

Figure 2. Polyurethane foam (PUF) sampling cartridge (a) and PUF-Tenax® TA "sandwich" sampling cartridge (b).

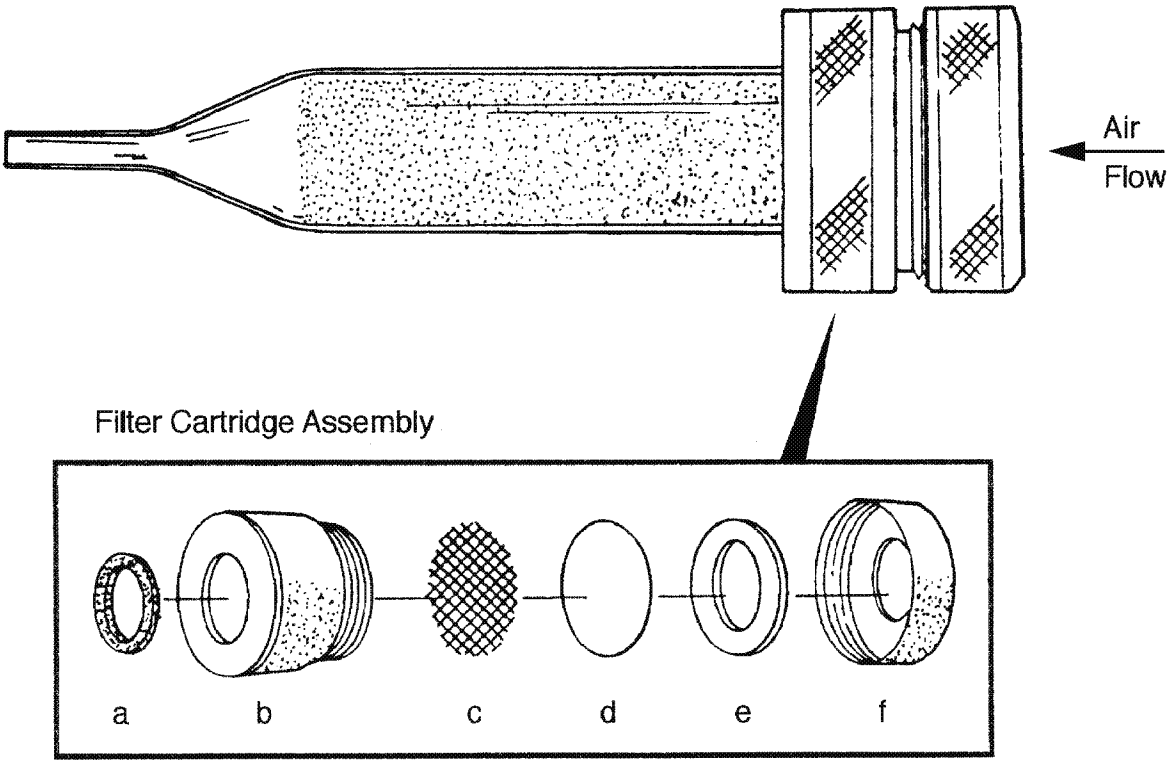

Figure 3. Open-face filter assembly attached to a PUF cartridge:
(a) Inner Viton® o-ring, (b) filter cartridge, (c) stainless steel screen, (d) quartz filter,
(e) filter ring, and (f) cartridge screw cap.

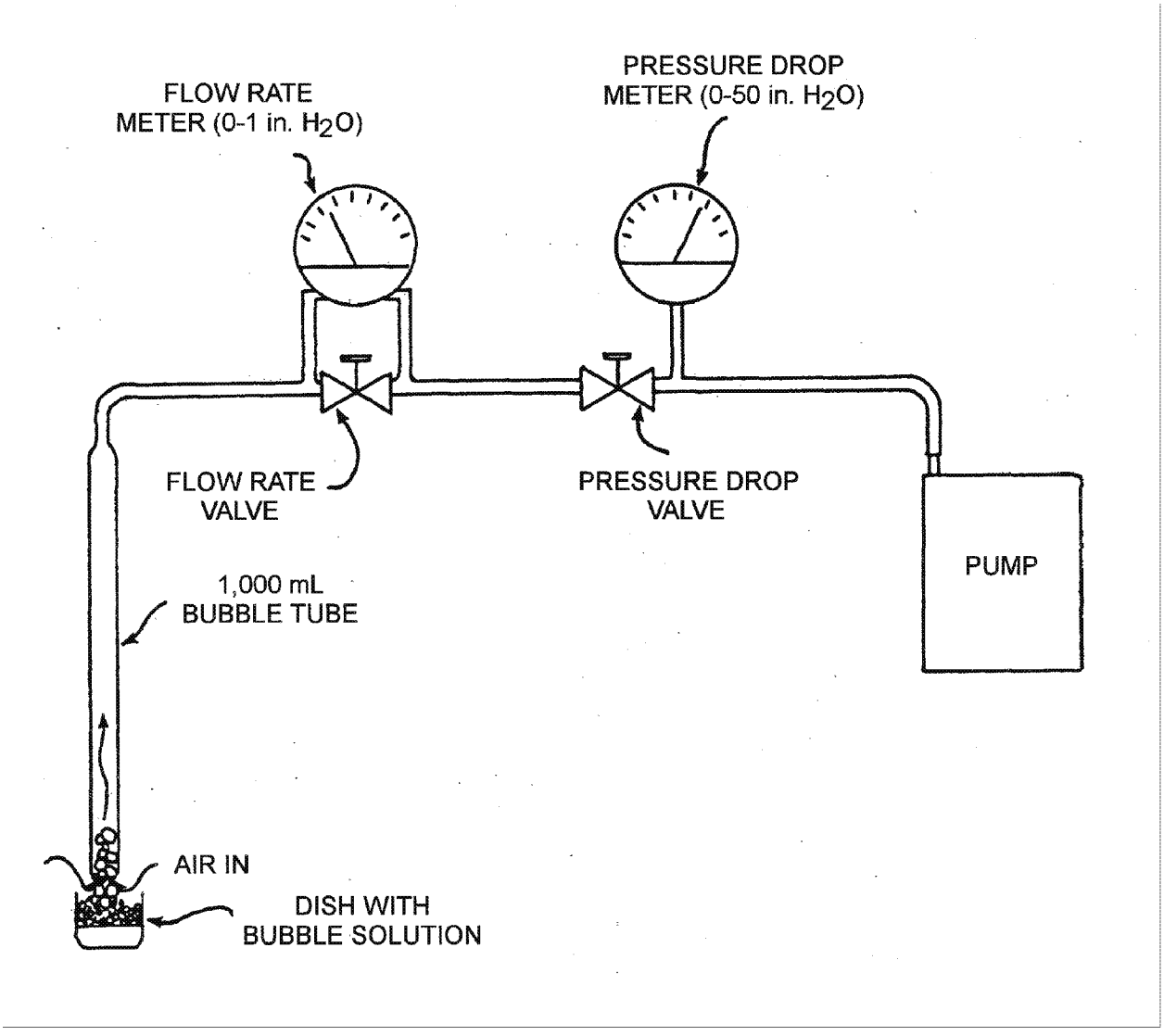

Figure 4. Calibration assembly for air sampler pump.

COMPENDIUM METHOD TO-10A
FIELD TEST DATA SHEET (FTDS)

I. GENERAL INFORMATION

PROJECT:_____ DATE(S) SAMPLED:_____
SITE:_____ TIME PERIOD SAMPLED:_____
LOCATION:_____ OPERATOR:_____
INSTRUMENT MODEL NO.:_____ CALIBRATED BY:_____
PUMP SERIAL NO.:_____ RAIN: ____YES _____NO

ADSORBENT CARTRIDGE INFORMATION:

	Cartridge 1	Cartridge 2	Cartridge 3	Cartridge 4
Type:	_____	_____	_____	_____
Adsorbent:	_____	_____	_____	_____
Serial No.:	_____	_____	_____	_____
Sample No.:	_____	_____	_____	_____

II. SAMPLING DATA

Cartridge Identification	Sampling Location	Ambient Temp., °F	Ambient Pressure, in Hg	Flow Rate (Q), mL/min		Sampling Period		Total Sampling Time, min.	Total Sample Volume, L
				Cartridge 1	Cartridge 2	Start	Stop		

III. FIELD AUDIT

	Cartridge 1	Cartridge 2	Cartridge 3	Cartridge 4
Audit Flow Check Within 10% of Set Point (Y/N)?	___ pre- _____ post-	_____ pre- _____ post-	_____ pre- _____ post-	_____ pre- _____ post-

CHECKED BY:_____

DATE:_____

Figure 5. Compendium Method TO-10A field test data sheet.

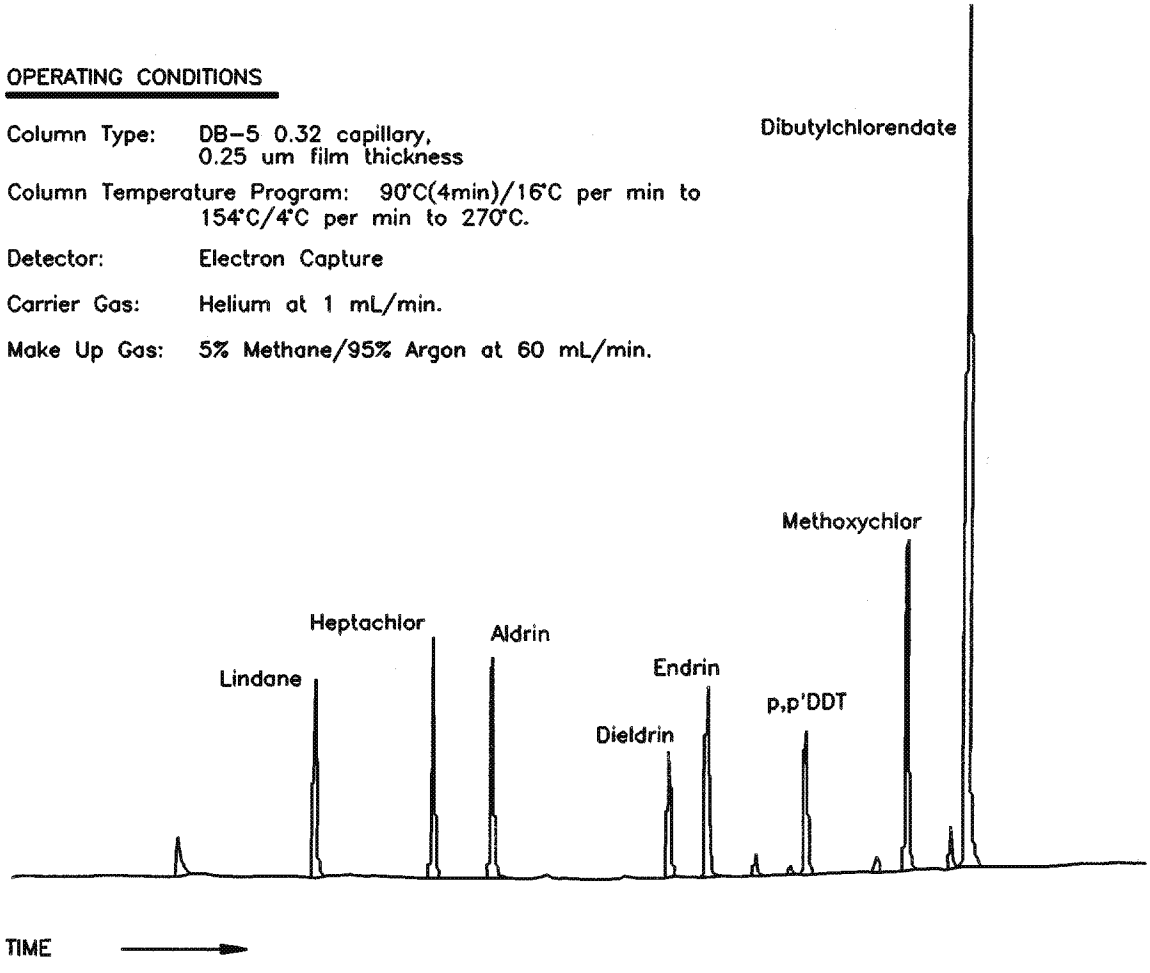

Figure 6. Chromatogram showing a mixture of single component pesticides determined by GC/ECD using a capillary column.

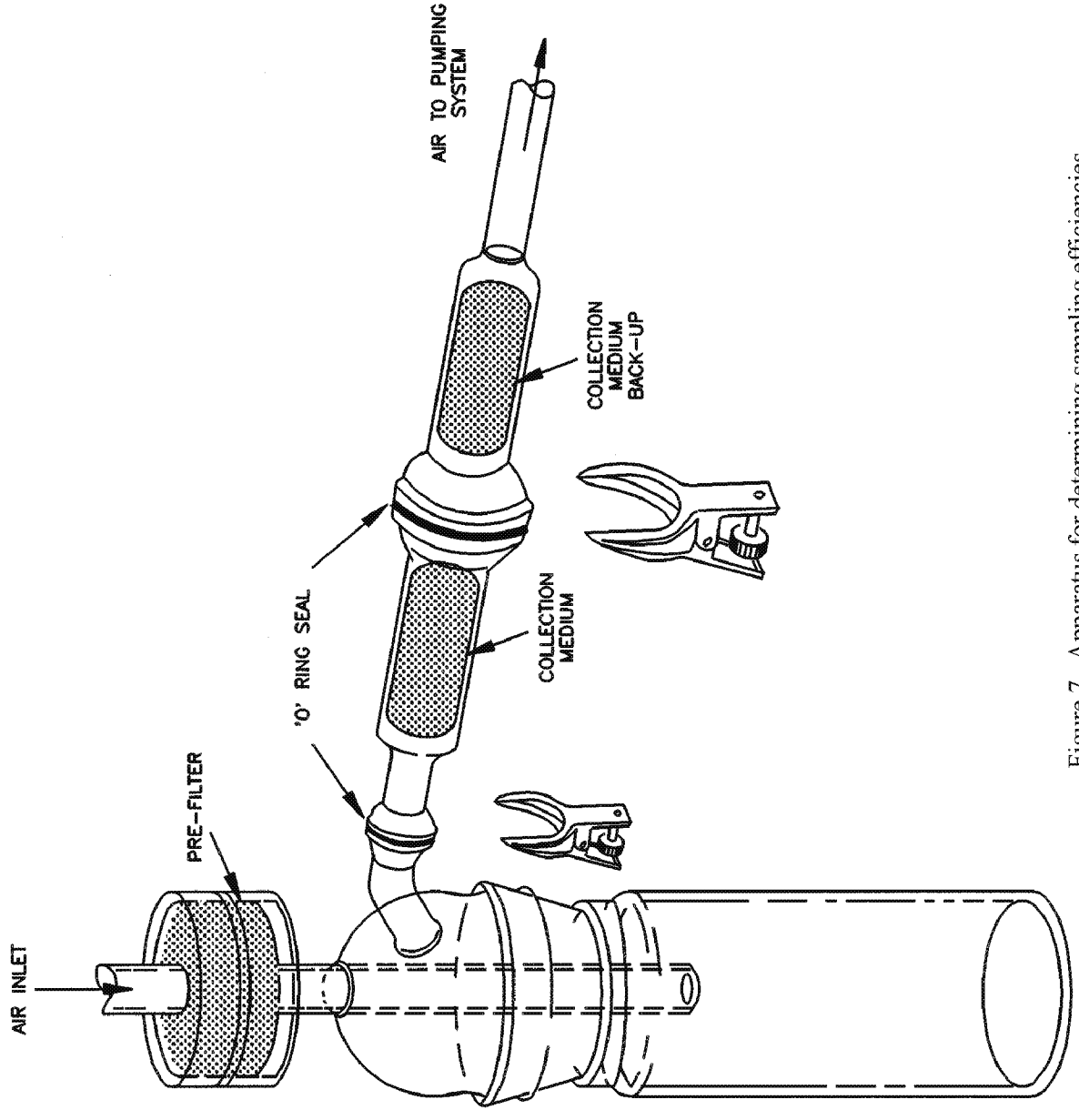

Figure 7. Apparatus for determining sampling efficiencies.

Appendix C

SW846 Method 8082

POLYCHLORINATED BIPHENYLS (PCBs) BY GAS CHROMATOGRAPHY

SW-846 is not intended to be an analytical training manual. Therefore, method procedures are written based on the assumption that they will be followed by individuals formally trained in at least the basic principles of chemical analysis and in the use of the subject technology.

In addition, SW-846 methods, with the exception of required method use for the analysis of method-defined parameters, are intended to be methods which contain general information on how to perform an analytical procedure or technique which a laboratory can use as a basic starting point for generating its own detailed standard operating procedure (SOP), either for its own general use or for a specific project application. The performance data included in this method are for guidance purposes only, and are not intended to be and must not be used as absolute QC acceptance criteria for purposes of laboratory accreditation.

1.0 SCOPE AND APPLICATION

1.1 This method may be used to determine the concentrations of polychlorinated biphenyls (PCBs) as Aroclors or as individual PCB congeners in extracts from solid, tissue, and aqueous matrices, using open-tubular, capillary columns with electron capture detectors (ECD) or electrolytic conductivity detectors (ELCD). The Aroclors and PCB congeners listed below have been determined by this method, using either a single- or dual column analysis system, and this method may be appropriate for additional congeners and Aroclors (see Sec. 1.4). The method also may be applied to other matrices such as oils and wipe samples, if appropriate sample extraction procedures are employed.

Compound	CAS Registry No.[a]	IUPAC #
Aroclor 1016	12674-11-2	-
Aroclor 1221	11104-28-2	-
Aroclor 1232	11141-16-5	-
Aroclor 1242	53469-21-9	-
Aroclor 1248	12672-29-6	-
Aroclor 1254	11097-69-1	-
Aroclor 1260	11096-82-5	-
2-Chlorobiphenyl	2051-60-7	1
2,3-Dichlorobiphenyl	16605-91-7	5
2,2',5-Trichlorobiphenyl	37680-65-2	18
2,4',5-Trichlorobiphenyl	16606-02-3	31
2,2',3,5'-Tetrachlorobiphenyl	41464-39-5	44
2,2',5,5'-Tetrachlorobiphenyl	35693-99-3	52
2,3',4,4'-Tetrachlorobiphenyl	32598-10-0	66
2,2',3,4,5'-Pentachlorobiphenyl	38380-02-8	87
2,2',4,5,5'-Pentachlorobiphenyl	37680-73-2	101
2,3,3',4',6-Pentachlorobiphenyl	38380-03-9	110
2,2',3,4,4',5'-Hexachlorobiphenyl	35065-28-2	138
2,2',3,4,5,5'-Hexachlorobiphenyl	52712-04-6	141

Compound	CAS Registry No.[a]	IUPAC #
2,2',3,5,5',6-Hexachlorobiphenyl	52663-63-5	151
2,2',4,4',5,5'-Hexachlorobiphenyl	35065-27-1	153
2,2',3,3',4,4',5-Heptachlorobiphenyl	35065-30-6	170
2,2',3,4,4',5,5'-Heptachlorobiphenyl	35065-29-3	180
2,2',3,4,4',5',6-Heptachlorobiphenyl	52663-69-1	183
2,2',3,4',5,5',6-Heptachlorobiphenyl	52663-68-0	187
2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl	40186-72-9	206

[a]Chemical Abstract Service Registry No.

1.2 Aroclors are multi-component mixtures. When samples contain more than one Aroclor, a higher level of analyst expertise is required to attain acceptable levels of qualitative and quantitative analysis. The same is true of Aroclors that have been subjected to environmental degradation ("weathering") or degradation by treatment technologies. Such weathered multi-component mixtures may have significant differences in peak patterns compared to those of Aroclor standards.

1.3 The seven Aroclors listed in Sec. 1.1 are those that are commonly specified in EPA regulations. The quantitation of PCBs as Aroclors is appropriate for many regulatory compliance determinations, but is particularly difficult when the Aroclors have been weathered by long exposure in the environment. Therefore, this method provides procedures for the determination of a selected group of the 209 possible PCB congeners, as another means to measure the concentrations of weathered Aroclors. The 19 PCB congeners listed above have been tested by this method and were chosen for testing because many of them represent congeners specific to the common Aroclor formulations (see Table 6). These 19 PCB congeners do not represent the co-planar PCBs or the other PCBs of greatest toxicological significance. **The analytical procedures for these 19 congeners may be appropriate for the analysis of other congeners not specifically included in this method and may be used as a template for the development of such a procedure.** However, all 209 PCB congeners cannot be separated using the GC columns and procedures described in this method. If this procedure is expanded to encompass other congeners, then the analyst must either document the resolution of the congeners in question, or establish procedures for reporting the results of coeluting congeners that are appropriate for the intended application.

1.4 The PCB congener approach potentially affords greater quantitative accuracy when PCBs are known to be present. As a result, this method may be used to determine Aroclors, some PCB congeners, or "total PCBs," depending on regulatory requirements and project needs. The congener method is of particular value in determining weathered Aroclors. However, analysts should use caution when using the congener method when regulatory requirements are based on Aroclor concentrations. Also, this method is not appropriate as currently written for the determination of the co-planar PCB congeners at the very low (sub part per trillion) concentrations sometimes needed for risk assessment purposes.

1.5 Compound identification based on single-column analysis should be confirmed on a second column, or should be supported by at least one other qualitative technique. This method describes analytical conditions for a second gas chromatographic column that can be used to confirm the measurements made with the primary column. GC/MS (e.g., Method 8270) is also recommended as a confirmation technique, if sensitivity permits (also see Sec. 11.11 of this method). GC/AED may also be used as a confirmation technique, if sensitivity permits (see Method 8085).

1.6 This method includes a dual-column option that describes a hardware configuration in which two GC columns are connected to a single injection port and to two separate detectors. The option allows one injection to be used for dual-column simultaneous analysis.

1.7 The analyst must select columns, detectors and calibration procedures most appropriate for the specific analytes of interest in a study. Matrix-specific performance data must be established and the stability of the analytical system and instrument calibration must be established for each analytical matrix (e.g., hexane solutions from sample extractions, diluted oil samples, etc.). Example chromatograms and GC conditions are provided as guidance.

1.8 Prior to employing this method, analysts are advised to consult the base method for each type of procedure that may be employed in the overall analysis (e.g., Methods 3500, 3600, and 8000) for additional information on quality control procedures, development of QC acceptance criteria, calculations, and general guidance. Analysts also should consult the disclaimer statement at the front of the manual and the information in Chapter Two for guidance on the intended flexibility in the choice of methods, apparatus, materials, reagents, and supplies, and on the responsibilities of the analyst for demonstrating that the techniques employed are appropriate for the analytes of interest, in the matrix of interest, and at the levels of concern.

In addition, analysts and data users are advised that, except where explicitly specified in a regulation, the use of SW-846 methods is *not* mandatory in response to Federal testing requirements. The information contained in this method is provided by EPA as guidance to be used by the analyst and the regulated community in making judgments necessary to generate results that meet the data quality objectives for the intended application.

1.9 Use of this method is restricted to use by, or under the supervision of, personnel appropriately experienced and trained in the use of gas chromatographs (GCs) and skilled in the interpretation of gas chromatograms. Each analyst must demonstrate the ability to generate acceptable results with this method.

2.0 SUMMARY OF METHOD

2.1 A measured volume or weight of sample is extracted using the appropriate matrix-specific sample extraction technique.

2.1.1 Aqueous samples may be extracted at neutral pH with methylene chloride using either Method 3510 (separatory funnel), Method 3520 (continuous liquid-liquid extractor), Method 3535 (solid-phase extraction), or other appropriate technique or solvents.

2.1.2 Solid samples may be extracted with hexane-acetone (1:1) or methylene chloride-acetone (1:1) using Method 3540 (Soxhlet), Method 3541 (automated Soxhlet), Method 3545 (pressurized fluid extraction), Method 3546 (microwave extraction), Method 3550 (ultrasonic extraction), Method 3562 (supercritical fluid extraction), or other appropriate technique or solvents.

2.1.3 Tissue samples may be extracted using Method 3562 (supercritical fluid extraction), or other appropriate technique. The extraction techniques for other solid matrices (see Sec. 2.1.2) may be appropriate for tissue samples.

2.2 Extracts for PCB analysis may be subjected to a sequential sulfuric acid/potassium permanganate cleanup (Method 3665) designed specifically for these analytes. This cleanup technique will remove (destroy) many single component organochlorine or organophosphorus pesticides. Therefore, this method is not applicable to the analysis of those compounds. Instead, use Method 8081.

2.3 After cleanup, the extract is analyzed by injecting a measured aliquot into a gas chromatograph equipped with either a narrow- or wide-bore fused-silica capillary column and either an electron capture detector (GC/ECD) or an electrolytic conductivity detector (GC/ELCD).

2.4 The chromatographic data may be used to determine the seven Aroclors in Sec. 1.1, selected individual PCB congeners, or total PCBs (see Secs. 11.8 and 11.9).

3.0 DEFINITIONS

Refer to Chapter One and the manufacturer's instructions for definitions that may be relevant to this procedure.

4.0 INTERFERENCES

4.1 Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and/or interferences to sample analysis. All of these materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks. Specific selection of reagents and purification of solvents by distillation in all-glass systems may be necessary. Refer to each method to be used for specific guidance on quality control procedures and to Chapter Four for general guidance on the cleaning of glassware. Also refer to Methods 3500, 3600, and 8000 for a discussion of interferences.

4.2 Interferences co-extracted from the samples will vary considerably from matrix to matrix. While general cleanup techniques are referenced or provided as part of this method, unique samples may require additional cleanup approaches to achieve desired degrees of discrimination and quantitation. Sources of interference in this method can be grouped into four broad categories, as follows:

4.2.1 Contaminated solvents, reagents, or sample processing hardware.

4.2.2 Contaminated GC carrier gas, parts, column surfaces, or detector surfaces.

4.2.3 Compounds extracted from the sample matrix to which the detector will respond, such as single-component chlorinated pesticides, including the DDT analogs (DDT, DDE, and DDD).

NOTE: A standard of the DDT analogs should be injected to determine which of the PCB or Aroclor peaks may be subject to interferences on the analytical columns used. There may be substantial DDT interference with the last major Aroclor 1254 peak in some soil and sediment samples.

4.2.4 Coelution of related analytes -- All 209 PCB congeners cannot be separated using the GC columns and procedures described in this method. If this procedure is expanded to encompass other congeners, then the analyst must either

document the resolution of the congeners in question or establish procedures for reporting the results of coeluting congeners that are appropriate for the intended application.

4.3 Interferences by phthalate esters introduced during sample preparation can pose a major problem in PCB determinations. Interferences from phthalate esters can best be minimized by avoiding contact with any plastic materials and checking all solvents and reagents for phthalate contamination.

4.3.1 Common flexible plastics contain varying amounts of phthalate esters which are easily extracted or leached from such materials during laboratory operations.

4.3.2 Exhaustive cleanup of solvents, reagents and glassware may be required to eliminate background phthalate ester contamination.

4.3.3 These materials can be removed prior to analysis using Method 3665 (sulfuric acid/permanganate cleanup).

4.4 Cross-contamination of clean glassware can routinely occur when plastics are handled during extraction steps, especially when solvent-wetted surfaces are handled. Glassware must be scrupulously cleaned.

4.4.1 Clean all glassware as soon as possible after use by rinsing with the last solvent used. This should be followed by detergent washing with hot water, and rinses with tap water and organic-free reagent water. Drain the glassware, and dry it in an oven at 130 °C for several hours, or rinse with methanol and drain. Store dry glassware in a clean environment.

CAUTION: Oven-drying of glassware used for PCB analysis can increase contamination because PCBs are readily volatilized in the oven and spread to other glassware. Therefore, exercise caution, and do not dry glassware from samples containing high concentrations of PCBs with glassware that may be used for trace analyses.

4.4.2 Other appropriate glassware cleaning procedures may be employed, such as using a muffle furnace at 430 °C for at least 30 min. However, analysts are advised not to place volumetric glassware in a muffle furnace, since the heat will burn off the markings on the glassware and may warp the glassware, changing its volume.

4.5 Sulfur (S_8) is readily extracted from soil samples and may cause chromatographic interferences in the determination of PCBs. Sulfur contamination should be expected with sediment samples. Sulfur can be removed through the use of Method 3660.

5.0 SAFETY

This method does not address all safety issues associated with its use. The laboratory is responsible for maintaining a safe work environment and a current awareness file of OSHA regulations regarding the safe handling of the chemicals listed in this method. A reference file of material safety data sheets (MSDSs) should be available to all personnel involved in these analyses.

6.0 EQUIPMENT AND SUPPLIES

The mention of trade names or commercial products in this manual is for illustrative purposes only, and does not constitute an EPA endorsement or exclusive recommendation for use. The products and instrument settings cited in SW-846 methods represent those products and settings used during method development or subsequently evaluated by the Agency. Glassware, reagents, supplies, equipment, and settings other than those listed in this manual may be employed provided that method performance appropriate for the intended application has been demonstrated and documented.

This section does not list common laboratory glassware (e.g., beakers and flasks).

6.1 Gas chromatograph -- An analytical system complete with gas chromatograph suitable for on-column and split-splitless injection and all necessary accessories including syringes, analytical columns, gases, electron capture detectors (ECD), and recorder/integrator or data system. Electrolytic conductivity detectors (ELCDs) may also be employed if appropriate for project needs. If the dual-column option is employed, the gas chromatograph must be equipped with two separate detectors.

6.2 GC columns

This method describes procedures for both single-column and dual-column analyses. The single-column approach involves one analysis to determine that a compound is present, followed by a second analysis to confirm the identity of the compound (Sec. 11.11 describes how GC/MS confirmation techniques may be employed). The single-column approach may employ either narrow-bore ($\leq$ 0.32-mm ID) columns or wide-bore (0.53-mm ID) columns. The dual-column approach generally employs a single injection that is split between two columns that are mounted in a single gas chromatograph. The dual-column approach generally employs wide-bore (0.53-mm ID) columns, but columns of other diameters may be employed if the analyst can demonstrate and document acceptable performance for the intended application. A third alternative is to employ dual columns mounted in a single GC, but with each column connected to a separate injector and a separate detector.

The columns listed in this section were the columns used in developing the method. The listing of these columns in this method is not intended to exclude the use of other columns that are available or that may be developed. Laboratories may use these columns or other columns provided that the laboratories document method performance data (e.g., chromatographic resolution, analyte breakdown, and sensitivity) that are appropriate for the intended application.

6.2.1 Narrow-bore columns for single-column analysis (use both columns to confirm compound identifications unless another confirmation technique such as GC/MS is employed). Narrow-bore columns should be installed in split/splitless (Grob-type) injectors.

6.2.1.1 30-m x 0.25-mm or 0.32-mm ID fused-silica capillary column chemically bonded with SE-54 (DB-5 or equivalent), 1-µm film thickness.

6.2.1.2 30-m x 0.25-mm ID fused-silica capillary column chemically bonded with 35 percent phenyl methylpolysiloxane (DB-608, SPB-608, or equivalent), 2.5 µm coating thickness, 1-µm film thickness.

6.2.2 Wide-bore columns for single-column analysis (use two of the three columns listed to confirm compound identifications unless another confirmation technique

such as GC/MS is employed). Wide-bore columns should be installed in 1/4-inch injectors, with deactivated liners designed specifically for use with these columns.

6.2.2.1 30-m x 0.53-mm ID fused-silica capillary column chemically bonded with 35 percent phenyl methylpolysiloxane (DB-608, SPB-608, RTx-35, or equivalent), 0.5-µm or 0.83-µm film thickness.

6.2.2.2 30-m x 0.53-mm ID fused-silica capillary column chemically bonded with 14% cyanopropylmethylpolysiloxane (DB-1701, or equivalent), 1.0-µm film thickness.

6.2.2.3 30-m x 0.53-mm ID fused-silica capillary column chemically bonded with SE-54 (DB-5, SPB-5, RTx-5, or equivalent), 1.5-µm film thickness.

6.2.3 Wide-bore columns for dual-column analysis -- The three pairs of recommended columns are listed below.

6.2.3.1 Column pair 1

30-m x 0.53-mm ID fused-silica capillary column chemically bonded with SE-54 (DB-5, SPB-5, RTx-5, or equivalent), 1.5-µm film thickness.

30-m x 0.53-mm ID fused-silica capillary column chemically bonded with 14% cyanopropylmethylpolysiloxane (DB-1701, or equivalent), 1.0-µm film thickness.

Column pair 1 is mounted in a press-fit Y-shaped glass 3-way union splitter (J&W Scientific, Catalog No. 705-0733) or a Y-shaped fused-silica connector (Restek, Catalog No. 20405), or equivalent.

NOTE: When connecting columns to a press-fit Y-shaped connector, a better seal may be achieved by first soaking the ends of the capillary columns in alcohol for about 10 sec to soften the polyimide coating.

6.2.3.2 Column pair 2

30-m x 0.53-mm ID fused-silica capillary column chemically bonded with SE-54 (DB-5, SPB-5, RTx-5, or equivalent), 0.83-µm film thickness.

30-m x 0.53-mm ID fused-silica capillary column chemically bonded with 14% cyanopropylmethylpolysiloxane (DB-1701, or equivalent), 1.0-µm film thickness.

Column pair 2 is mounted in an 8-in. deactivated glass injection tee (Supelco, Catalog No. 2-3665M), or equivalent.

6.2.3.3 Column pair 3

30-m x 0.53-mm ID fused-silica capillary column chemically bonded with SE-54 (DB-5, SPB-5, RTx-5, or equivalent), 1.5-µm film thickness.

30-m x 0.53-mm ID fused-silica capillary column chemically bonded with 35 percent phenyl methylpolysiloxane (HP-608, DB-608, SPB-608, RTx-35, or equivalent), 0.5-µm film thickness.

Column pair 3 is mounted in separate injectors and separate detectors.

6.3 Column rinsing kit -- Bonded-phase column rinse kit (J&W Scientific, Catalog No. 430-3000), or equivalent.

6.4 Volumetric flasks -- 10-mL and 25-mL, for preparation of standards.

6.5 Analytical balance, capable of weighing to 0.0001 g.

7.0 REAGENTS AND STANDARDS.

7.1 Reagent-grade or pesticide-grade chemicals must be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. Reagents should be stored in glass to prevent the leaching of contaminants from plastic containers.

NOTE: Store the standard solutions (stock, composite, calibration, internal, and surrogate) at ≤6 °C in polytetrafluoroethylene (PTFE)-sealed containers in the dark. When a lot of standards is prepared, aliquots of that lot should be stored in individual small vials. All stock standard solutions must be replaced after one year, or sooner if routine QC (see Sec. 9.0) indicates a problem. All other standard solutions must be replaced after six months, or sooner if routine QC (see Sec. 9.0) indicates a problem.

7.2 Solvents used in the extraction and cleanup procedures (appropriate 3500 and 3600 series methods) include *n*-hexane, diethyl ether, methylene chloride, acetone, ethyl acetate, and isooctane (2,2,4-trimethylpentane) and the solvents must be exchanged to *n*-hexane or isooctane prior to analysis. Therefore, *n*-hexane and isooctane will be required in this procedure. All solvents should be pesticide grade in quality or equivalent, and each lot of solvent should be determined to be free of phthalates.

7.3 The following solvents may be necessary for the preparation of standards. All solvent lots must be pesticide grade in quality or equivalent and should be determined to be free of phthalates.

7.3.1 Acetone, $(CH_3)_2CO$

7.3.2 Toluene, $C_6H_5CH_3$

7.4 Organic-free reagent water -- All references to water in this method refer to organic-free reagent water as defined in Chapter One.

7.5 Standard solutions

The following sections describe the preparation of stock, intermediate, and working standards for the compounds of interest. This discussion is provided as an example, and other approaches and concentrations of the target compounds may be used, as appropriate for the intended application. See Method 8000 for additional information on the preparation of calibration standards.

7.6 Stock standard solutions (1000 mg/L) -- May be prepared from pure standard materials or can be purchased as certified solutions.

7.6.1 Prepare stock standard solutions by accurately weighing 0.0100 g of pure compound. Dissolve the compound in isooctane or hexane and dilute to volume in a 10-mL volumetric flask. If compound purity is 96 percent or greater, the weight can be used without correction to calculate the concentration of the stock standard solution.

7.6.2 Commercially-prepared stock standard solutions may be used at any concentration if they are certified by the manufacturer or by an independent source.

7.7 Calibration standards for Aroclors

7.7.1 A standard containing a mixture of Aroclor 1016 and Aroclor 1260 will include many of the peaks represented in the other five Aroclor mixtures. As a result, a multi-point initial calibration employing a mixture of Aroclors 1016 and 1260 at five concentrations should be sufficient to demonstrate the linearity of the detector response without the necessity of performing multi-point initial calibrations for each of the seven Aroclors. In addition, such a mixture can be used as a standard to demonstrate that a sample does not contain peaks that represent any one of the Aroclors. This standard can also be used to determine the concentrations of either Aroclor 1016 or Aroclor 1260, should they be present in a sample.

Prepare a minimum of five calibration standards containing equal concentrations of both Aroclor 1016 and Aroclor 1260 by dilution of the stock standard with isooctane or hexane. The concentrations should correspond to the expected range of concentrations found in real samples and should bracket the linear range of the detector. See Method 8000 for additional information regarding the preparation of calibration standards.

7.7.2 Single standards of each of the other five Aroclors are required to aid the analyst in pattern recognition. Assuming that the Aroclor 1016/1260 standards described in Sec. 7.7.1 have been used to demonstrate the linearity of the detector, these single standards of the remaining five Aroclors also may be used to determine the calibration factor for each Aroclor when a linear calibration model through the origin is chosen (see Sec. 11.4). Prepare a standard for each of the other Aroclors. The concentrations should generally correspond to the mid-point of the linear range of the detector, but lower concentrations may be employed at the discretion of the analyst based on project requirements.

7.7.3 Other standards (e.g., other Aroclors) and other calibration approaches (e.g., non-linear calibration for individual Aroclors) may be employed to meet project needs. When the nature of the PCB contamination is already known, use standards of those particular Aroclors. See Method 8000 for information on non-linear calibration approaches.

7.8 Calibration standards for PCB congeners

7.8.1 If results are to be determined for individual PCB congeners, then standards for the pure congeners must be prepared. The table in Sec. 1.1 lists 19 PCB congeners that have been tested by this method along with the IUPAC numbers designating these congeners. This procedure may be appropriate for other congeners as well, but the analyst must either document the resolution of the congeners in question or establish procedures for reporting the results of coeluting congeners that are appropriate for the intended application.

7.8.2 Stock standards may be prepared in a fashion similar to that described for the Aroclor standards, or may be purchased as commercially-prepared solutions. Stock standards should be used to prepare a minimum of five concentrations by dilution of the stock standard with isooctane or hexane. The concentrations should correspond to the expected range of concentrations found in real samples and should bracket the linear range of the detector.

7.9 Internal standard

7.9.1 When PCB congeners are to be determined, the use of an internal standard is highly recommended. Decachlorobiphenyl may be used as an internal standard, added to each sample extract prior to analysis, and included in each of the initial calibration standards.

7.9.2 When PCBs are to be determined as Aroclors, an internal standard is typically not used, and decachlorobiphenyl is employed as a surrogate (see Sec. 7.10).

7.9.3 When decachlorobiphenyl is an analyte of interest, as in some PCB congener analyses, see Sec. 7.10.3.

7.10 Surrogate standards

The performance of the method should be monitored using surrogate compounds. Surrogate standards are added to all samples, method blanks, matrix spikes, and calibration standards. The choice of surrogate compounds will depend on analysis mode chosen, e.g., Aroclors or congeners. The following compounds are recommended as surrogates. Other surrogates may be used, provided that the analyst can demonstrate and document performance appropriate for the data quality needs of the particular application.

7.10.1 When PCBs are to be determined as Aroclors, decachlorobiphenyl may be used as a surrogate, and is added to each sample prior to extraction. Prepare a solution of decachlorobiphenyl in acetone. The recommended spiking solution concentration is 5 mg/L. Tetrachloro-*m*-xylene also may be used as a surrogate for Aroclor analysis. If used, the recommended spiking solution concentration is 5 mg/L in acetone. (Other surrogate concentrations may be used, as appropriate for the intended application.)

7.10.2 When PCB congeners are to be determined, decachlorobiphenyl is recommended for use as an internal standard, and therefore it cannot also be used as a surrogate. Tetrachloro-*m*-xylene may be used as a surrogate for PCB congener analysis. The recommended spiking solution concentration is 5 mg/L in acetone. (Other surrogate concentrations may be used, as appropriate for the intended application.)

7.10.3 If decachlorobiphenyl is a target congener for the analysis, 2,2',4,4',5,5'-hexabromobiphenyl may be used as an internal standard or a surrogate.

7.11 DDT analog standard -- Used to determine if the commonly found DDT analogs (DDT, DDE, and DDD) elute at the same retention times as any of the target analytes (congeners or Aroclors). A single standard containing all three compounds should be sufficient. The concentration of the standard is left to the judgement of the analyst.

8.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1 See the introductory material to Chapter Four, "Organic Analytes."

8.2 Extracts should be stored under refrigeration in the dark and should be analyzed within 40 days of extraction.

NOTE: The holding time above is a recommendation. PCBs are very stable in a variety of matrices, and holding times under the conditions listed above may be as long as a year.

9.0 QUALITY CONTROL

9.1 Refer to Chapter One for guidance on quality assurance (QA) and quality control (QC) protocols. When inconsistencies exist between QC guidelines, method-specific QC criteria take precedence over both technique-specific criteria and those criteria given in Chapter One, and technique-specific QC criteria take precedence over the criteria in Chapter One. Any effort involving the collection of analytical data should include development of a structured and systematic planning document, such as a Quality Assurance Project Plan (QAPP) or a Sampling and Analysis Plan (SAP), which translates project objectives and specifications into directions for those that will implement the project and assess the results. Each laboratory should maintain a formal quality assurance program. The laboratory should also maintain records to document the quality of the data generated. All data sheets and quality control data should be maintained for reference or inspection.

9.2 Refer to Method 8000 for specific determinative method QC procedures. Refer to Method 3500 for QC procedures to ensure the proper operation of the various sample preparation techniques. If an extract cleanup procedure is performed, refer to Method 3600 for the appropriate QC procedures. Any more specific QC procedures provided in this method will supersede those noted in Methods 8000, 3500, or 3600.

9.3 Quality control procedures necessary to evaluate the GC system operation are found in Method 8000 and include evaluation of retention time windows, calibration verification and chromatographic analysis of samples.

9.3.1 Include a calibration standard after each group of 20 samples (it is *recommended* that a calibration standard be included after every 10 samples to minimize the number of repeat injections) in the analysis sequence as a calibration check. Thus, injections of method blank extracts, matrix spike samples, and other non-standards are counted in the total. Solvent blanks, injected as a check on cross-contamination, need not be counted in the total. The response factors for the calibration should be within ±20 percent of the initial calibration (see Sec. 11.6.2). When this continuing calibration is out of this acceptance window, the laboratory should stop analyses and take corrective action.

9.3.2 Whenever quantitation is accomplished using an internal standard, internal standards must be evaluated for acceptance. The measured area of the internal standard must be no more than 50 percent different from the average area calculated during initial calibration. When the internal standard peak area is outside the limit, all samples that fall outside the QC criteria must be reanalyzed. The retention times of the internal standards must also be evaluated. A retention time shift of >30 sec necessitates reanalysis of the affected sample.

9.4 Initial demonstration of proficiency

9.4.1 Each laboratory must demonstrate initial proficiency with each sample preparation and determinative method combination it utilizes, by generating data of acceptable accuracy and precision for target analytes in a clean matrix. If an autosampler is used to perform sample dilutions, before using the autosampler to dilute samples, the laboratory should satisfy itself that those dilutions are of equivalent or better accuracy than is achieved by an experienced analyst performing manual dilutions. The laboratory must also repeat the demonstration of proficiency whenever new staff members are trained or significant changes in instrumentation are made. See Method 8000 for information on how to accomplish a demonstration of proficiency.

9.4.2 It is suggested that the QC reference sample concentrate (as discussed in Methods 8000 and Method 3500) contain PCBs as Aroclors at 10-50 mg/L in the concentrate for water samples, or PCBs as congeners at the same concentrations. A 1-mL volume of this concentrate spiked into 1 L of reagent water will result in a sample concentration of 10-50 µg/L. If Aroclors are not expected in samples from a particular source, then prepare the QC reference samples with a mixture of Aroclors 1016 and 1260. However, when specific Aroclors are known to be present or expected in samples, the specific Aroclors should be used for the QC reference sample. See Method 8000 for additional information on how to accomplish this demonstration. Other concentrations may be used, as appropriate for the intended application.

9.4.3 Calculate the average recovery and the standard deviation of the recoveries of the analytes in each of the four QC reference samples. Refer to Method 8000 for procedures for evaluating method performance.

9.5 Initially, before processing any samples, the analyst should demonstrate that all parts of the equipment in contact with the sample and reagents are interference-free. This is accomplished through the analysis of a method blank. As a continuing check, each time samples are extracted, cleaned up, and analyzed, and when there is a change in reagents, a method blank should be prepared and analyzed for the compounds of interest as a safeguard against chronic laboratory contamination. If a peak is observed within the retention time window of any analyte that would prevent the determination of that analyte, determine the source and eliminate it, if possible, before processing the samples. The blanks should be carried through all stages of sample preparation and analysis. When new reagents or chemicals are received, the laboratory should monitor the preparation and/or analysis blanks associated with samples for any signs of contamination. It is not necessary to test every new batch of reagents or chemicals prior to sample preparation if the source shows no prior problems. However, if reagents are changed during a preparation batch, separate blanks need to be prepared for each set of reagents.

9.6 Sample quality control for preparation and analysis

The laboratory must also have procedures for documenting the effect of the matrix on method performance (precision, accuracy, method sensitivity). At a minimum, this should include the analysis of QC samples including a method blank, a matrix spike, a duplicate, and a laboratory control sample (LCS) in each analytical batch and the addition of surrogates to each field sample and QC sample when surrogates are used. Any method blanks, matrix spike samples, and replicate samples should be subjected to the same analytical procedures (Sec. 11.0) as those used on actual samples.

9.6.1 Documenting the effect of the matrix should include the analysis of at least one matrix spike and one duplicate unspiked sample or one matrix spike/matrix spike

duplicate pair. The decision on whether to prepare and analyze duplicate samples or a matrix spike/matrix spike duplicate must be based on a knowledge of the samples in the sample batch. If samples are expected to contain target analytes, then laboratories may use one matrix spike and a duplicate analysis of an unspiked field sample. If samples are not expected to contain target analytes, the laboratories should use a matrix spike and matrix spike duplicate pair, spiked with the Aroclor 1016/1260 mixture. However, when specific Aroclors are known to be present or expected in samples, the specific Aroclors should be used for spiking. Consult Method 8000 for information on developing acceptance criteria for the MS/MSD.

9.6.2 A laboratory control sample (LCS) should be included with each analytical batch. The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. The LCS is spiked with the same analytes at the same concentrations as the matrix spike, when appropriate. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix. Consult Method 8000 for information on developing acceptance criteria for the LCS.

9.6.3 Also see Method 8000 for the details on carrying out sample quality control procedures for preparation and analysis. In-house acceptance criteria for evaluating method performance should be developed using the guidance found in Method 8000.

9.7 Surrogate recoveries

If surrogates are used, the laboratory should evaluate surrogate recovery data from individual samples versus the surrogate control limits developed by the laboratory. See Method 8000 for information on evaluating surrogate data and developing and updating surrogate limits. Procedures for evaluating the recoveries of multiple surrogates and the associated corrective actions should be defined in an approved project plan.

9.8 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

10.0 CALIBRATION AND STANDARDIZATION

See Sec. 11.0 for information on calibration and standardization.

11.0 PROCEDURE

11.1 Sample extraction

11.1.1 Refer to Chapter Two and Method 3500 for guidance in choosing the appropriate extraction procedure. In general, water samples are extracted at a neutral pH with methylene chloride using a separatory funnel (Method 3510), a continuous liquid-liquid extractor (Method 3520), solid-phase extraction (Method 3535), or other appropriate technique. Solid samples are extracted with hexane-acetone (1:1) or methylene chloride-acetone (1:1) using one of the Soxhlet extraction methods (Method 3540 or 3541), pressurized fluid extraction (Method 3545), microwave extraction (Method 3546),

ultrasonic extraction (Method 3550), supercritical fluid extraction (Method 3562), or other appropriate technique or solvents. Tissue samples are extracted using supercritical fluid extraction (Method 3562) or other appropriate technique.

NOTE: The use of hexane-acetone generally reduces the amount of interferences that are extracted and improves signal-to-noise.

The choice of extraction solvent and procedure will depend on the analytes of interest. No single solvent or extraction procedure is universally applicable to all analyte groups and sample matrices. The analyst *must* demonstrate adequate performance for the analytes of interest, at the levels of interest, for any solvent system and extraction procedure employed, *including* those specifically listed in this method. At a minimum, such a demonstration will encompass the initial demonstration of proficiency described in Method 3500, using a clean reference matrix. Each new sample type must be spiked with the compounds of interest to determine the percent recovery. Method 8000 describes procedures that may be used to develop performance criteria for such demonstrations as well as for matrix spike and laboratory control sample results.

11.1.2 Reference materials, field-contaminated samples, or spiked samples should be used to verify the applicability of the selected extraction technique to each new sample type. Such samples should contain or be spiked with the compounds of interest in order to determine the percent recovery and the limit of detection for that sample type (see Chapter One). When other materials are not available and spiked samples are used, they should be spiked with the analytes of interest, either specific Aroclors or PCB congeners. When the presence of specific Aroclors is not anticipated, the Aroclor 1016/1260 mixture may be an appropriate choice for spiking. See Methods 3500 and 8000 for guidance on demonstration of initial method proficiency as well as guidance on matrix spikes for routine sample analysis.

11.1.3 The extraction techniques for solids may be applicable to wipe samples and other sample matrices not addressed in Sec. 11.1.1. The analysis of oil samples may need special sample preparation procedures that are not described here. Analysts should follow the steps described in Sec. 11.1.2 to verify the applicability of the sample preparation and extraction techniques for matrices such as wipes and oils.

11.2 Extract cleanup

Cleanup procedures may not be necessary for a relatively clean sample matrix, but most extracts from environmental and waste samples will require additional preparation before analysis. The specific cleanup procedure used will depend on the nature of the sample to be analyzed and the data quality objectives for the measurements. Refer to Methods 3600, 3660 and 3665 for general guidance on extract cleanup.

11.3 GC conditions

This method allows the analyst to choose between a single-column or a dual-column configuration in the injector port. The columns listed in this section were the columns used to develop the method performance data. Listing these columns in this method is not intended to exclude the use of other columns that are available or that may be developed. Wide-bore or narrow-bore columns may be used with either option. Laboratories may use either the columns listed in this method or other capillary columns or columns of other dimensions, provided that the laboratories document method performance data (e.g., chromatographic resolution, analyte breakdown, and sensitivity) that are appropriate for the intended application.

11.3.1 Single-column analysis

This capillary GC/ECD method allows the analyst the option of using 0.25-mm or 0.32-mm ID capillary columns (narrow-bore) or 0.53-mm ID capillary columns (wide-bore). Narrow-bore columns generally provide greater chromatographic resolution than wide-bore columns, although narrow-bore columns have a lower sample capacity. As a result, narrow-bore columns may be more suitable for relatively clean samples or for extracts that have been prepared with one or more of the clean-up options referenced in the method. Wide-bore columns (0.53-mm ID) may be more suitable for more complex environmental and waste matrices. However, the choice of the appropriate column diameter is left to the professional judgement of the analyst.

11.3.2 Dual-column analysis

The dual-column/dual-detector approach recommends the use of two 30-m x 0.53-mm ID fused-silica open-tubular columns of different polarities, thus, different selectivities towards the target analytes. The columns may be connected to an injection tee and separate electron capture detectors, or to both separate injectors and separate detectors. However, the choice of the appropriate column dimensions is left to the professional judgement of the analyst.

11.3.3 GC temperature programs and flow rates

11.3.3.1 Table 1 lists suggested GC operating conditions for the analysis of PCBs as Aroclors for single-column analysis, using either narrow-bore or wide-bore capillary columns. Table 2 lists suggested GC operating conditions for the dual-column analysis. Use the conditions in these tables as guidance and establish the GC temperature program and flow rate necessary to separate the analytes of interest.

11.3.3.2 When determining PCBs as congeners, difficulties may be encountered with coelution of congener 153 and other sample components. When determining PCBs as Aroclors, chromatographic conditions should be adjusted to give adequate separation of the characteristic peaks in each Aroclor (see Sec. 11.4.6).

11.3.3.3 Tables 3 and 4 summarize example retention times of up to 73 Aroclor peaks determined during dual-column analysis using the operating conditions listed in Table 2. These retention times are provided as guidance as to what may be achieved using the GC columns, temperature programs, and flow rates described in this method. Each laboratory must determine retention times and retention time windows for their specific application of the method. Note that the peak numbers used in these tables are *not* the IUPAC congener numbers, but represent the elution order of the peaks on these GC columns.

11.3.3.4 Once established, the same operating conditions must be used for the analysis of samples and standards.

11.4 Calibration

11.4.1 Prepare calibration standards using the procedures in Sec. 7.0. Refer to Method 8000 and Sec. 9.3 for proper calibration techniques for both initial calibration and calibration verification. When PCBs are to be determined as congeners, the use of internal standard calibration is highly recommended. Therefore, the calibration standards

must contain the internal standard (see Sec. 7.9) at the same concentration as the sample extracts. When PCBs are to be determined as Aroclors, external standard calibration is generally used.

NOTE: Because of the sensitivity of the electron capture detector, always clean the injection port and column prior to performing the initial calibration.

11.4.2 When PCBs are to be quantitatively determined as congeners, an initial multi-point calibration must be performed that includes standards for all the target analytes (congeners). See Method 8000 for details on calibration options.

11.4.3 When PCBs are to be quantitatively determined as Aroclors, the initial calibration consists of two parts, described below.

11.4.3.1 As noted in Sec. 7.7.1, a standard containing a mixture of Aroclor 1016 and Aroclor 1260 will include many of the peaks represented in the other five Aroclor mixtures. Thus, such a standard may be used to demonstrate the linearity of the detector and that a sample does not contain peaks that represent any one of the Aroclors. This standard can also be used to determine the concentrations of either Aroclor 1016 or Aroclor 1260, should they be present in a sample. Therefore, an initial multi-point calibration is performed using the mixture of Aroclors 1016 and 1260 described in Sec. 7.7.1. See Method 8000 for guidance on the use of linear and non-linear calibrations.

11.4.3.2 Standards of the other five Aroclors are necessary for pattern recognition. When employing the traditional model of a linear calibration through the origin, these standards are also used to determine a single-point calibration factor for each Aroclor, assuming that the Aroclor 1016/1260 mixture in Sec. 11.4.3.1 has been used to describe the detector response. The standards for these five Aroclors should be analyzed before the analysis of any samples, and may be analyzed before or after the analysis of the five 1016/1260 standards in Sec. 11.4.3.1. For non-linear calibrations, see Sec. 11.4.3.3.

11.4.3.3 In situations where only a few Aroclors are of interest for a specific project, the analyst may employ a multi-point initial calibration of each of the Aroclors of interest (e.g., five standards of Aroclor 1232 if this Aroclor is of concern and linear calibration is employed) and not use the 1016/1260 mixture described in Sec. 11.4.3.1 or the pattern recognition standards described in 11.4.3.2. When non-linear calibration models are employed, more than five standards of each Aroclor of interest will be needed to adequately describe the detector response (see Method 8000).

11.4.4 Establish the GC operating conditions appropriate for the configuration (single-column or dual column, Sec. 11.3), using Tables 1 or 2 as guidance. Optimize the instrumental conditions for resolution of the target compounds and sensitivity. A final temperature of between 240 °C and 275 °C may be needed to elute decachlorobiphenyl. The use of injector pressure programming will improve the chromatography of late eluting peaks.

NOTE: Once established, the same operating conditions must be used for both calibrations and sample analyses.

11.4.5 A 2-µL injection of each calibration standard is recommended. Other injection volumes may be employed, provided that the analyst can demonstrate adequate sensitivity for the compounds of interest.

11.4.6 Record the peak area (or height) for each congener or each characteristic Aroclor peak to be used for quantitation.

11.4.6.1 A minimum of 3 peaks must be chosen for each Aroclor, and preferably 5 peaks. The peaks must be characteristic of the Aroclor in question. Choose peaks in the Aroclor standards that are at least 25% of the height of the largest Aroclor peak. For each Aroclor, the set of 3 to 5 peaks should include at least one peak that is unique to that Aroclor. Use at least five peaks for the Aroclor 1016/1260 mixture, none of which should be found in both of these Aroclors.

11.4.6.2 Late-eluting Aroclor peaks are generally the most stable in the environment. Table 5 lists diagnostic peaks in each Aroclor, along with example retention times on two GC columns suitable for single-column analysis. Table 6 lists 13 specific PCB congeners found in Aroclor mixtures. Table 7 lists PCB congeners with example retention times on a DB-5 wide-bore GC column. Use these tables as guidance in choosing the appropriate peaks. Each laboratory must determine retention times and retention time windows for their specific application of the method.

11.4.7 When determining PCB congeners by the internal standard procedure, calculate the response factor (RF) for each congener in the calibration standards relative to the internal standard, decachlorobiphenyl, using the equation that follows.

$$RF = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

where:

A_s = Peak area (or height) of the analyte or surrogate.
A_{is} = Peak area (or height) of the internal standard.
C_s = Concentration of the analyte or surrogate, in µg/L.
C_{is} = Concentration of the internal standard, in µg/L.

11.4.8 When determining PCBs as Aroclors by the external standard technique, calculate the calibration factor (CF) for each characteristic Aroclor peak in each of the initial calibration standards (from either Sec. 11.4.3.1 or 11.4.3.2) using the equation below.

$$CF = \frac{\text{Peak Area (or Height) in the Standard}}{\text{Total Mass of the Standard Injected (in nanograms)}}$$

Using the equation above, a calibration factor will be determined for each characteristic peak, using the total mass of the Aroclor injected. These individual calibration factors are used to quantitate sample results by applying the factor for each individual peak to the area of that peak, as described in Sec. 11.9.

For a five-point calibration, five sets of calibration factors will be generated for the Aroclor 1016/1260 mixture, each set consisting of the calibration factors for each of the five (or more) peaks chosen for this mixture, e.g., there will be at least 25 separate calibration factors for the mixture. The single standard for each of the other Aroclors (see Sec. 11.4.3.1) will generate at least three calibration factors, one for each selected peak.

If a non-linear calibration model is employed, as described in Method 8000, then additional standards containing each Aroclor of interest will be employed, with a corresponding increase in the total number of calibration factors.

11.4.9 The response factors or calibration factors from the initial calibration are used to evaluate the linearity of the initial calibration, if a linear calibration model is used. This involves the calculation of the mean response or calibration factor, the standard deviation, and the relative standard deviation (RSD) for each congener or Aroclor peak.

When the Aroclor 1016/1260 mixture is used to demonstrate the detector response, the linear calibration models must be applied to the other five Aroclors for which only single standards are analyzed. If multi-point calibration is performed for individual Aroclors (see Sec. 11.4.3.3), use the calibration factors from those standards to evaluate linearity.

See Method 8000 for the specifics of the evaluation of the linearity of the calibration and guidance on performing non-linear calibrations. In general, non-linear calibrations also will consider each characteristic Aroclor peak separately.

11.5 Retention time windows

Absolute retention times are generally used for compound identification. When absolute retention times are used, retention time windows are crucial to the identification of target compounds, and should be established by one of the approaches described in Method 8000. Retention time windows are established to compensate for minor shifts in absolute retention times as a result of sample loadings and normal chromatographic variability. The width of the retention time window should be carefully established to minimize the occurrence of both false positive and false negative results. Tight retention time windows may result in false negatives and/or may cause unnecessary reanalysis of samples when surrogates or spiked compounds are erroneously not identified. Overly wide retention time windows may result in false positive results that cannot be confirmed upon further analysis. Analysts should consult Method 8000 for the details of establishing retention time windows. Other approaches to compound identification may be employed, provided that the analyst can demonstrate and document that the approaches are appropriate for the intended application. When PCBs are determined as congeners by an internal standard technique, absolute retention times may be used in conjunction with relative retention times (relative to the internal standard).

When conducting either Aroclor or congener analysis, it is important to determine that common single-component pesticides such as DDT, DDD, and DDE do not elute at the same retention times as the target congeners. There may be substantial DDT interference with the last major Aroclor 1254 peak in some soil and sediment samples. Therefore, in conjunction with determining the retention time windows of the congeners, the analyst should analyze a standard containing the DDT analogs. This standard need only be analyzed when the retention time

windows are determined. It is not considered part of the routine initial calibration or calibration verification steps in the method, nor are there any performance criteria associated with the analysis of this standard.

If Aroclor analysis is performed and any of the DDT analogs elute at the same retention time as an Aroclor peak that was chosen for use in quantitation (see Sec. 11.4.6), then the analyst must either adjust the GC conditions to achieve better resolution, or choose another peak that is characteristic of that Aroclor and does not correspond to a peak from a DDT analog. If PCB congener analysis is performed and any of the DDT analogs elute at the same retention time as a PCB congener of interest, then the analyst must adjust the GC conditions to achieve better resolution.

11.6 Gas chromatographic analysis of sample extracts

11.6.1 The same GC operating conditions used for the initial calibration must be employed for the analysis of samples.

11.6.2 Verify calibration at least once each 12-hr shift by injecting calibration verification standards prior to conducting any sample analyses. A calibration standard must also be injected at intervals of not less than once every twenty samples (after every 10 samples is recommended to minimize the number of samples requiring reinjection when QC limits are exceeded) and at the end of the analysis sequence. For Aroclor analyses, the calibration verification standard should be a mixture of Aroclor 1016 and Aroclor 1260. The calibration verification process does not *require* analysis of the other Aroclor standards used for pattern recognition, but the analyst may wish to include a standard for one of these Aroclors after the 1016/1260 mixture used for calibration verification throughout the analytical sequence.

11.6.2.1 The calibration factor for each analyte calculated from the calibration verification standard (CF_v) should not exceed a difference of more than ±20 percent when compared to the mean calibration factor from the initial calibration curve. If a calibration approach other than the RSD method has been employed for the initial calibration (e.g., a linear model not through the origin, a non-linear calibration model, etc.), consult Method 8000 for the specifics of calibration verification.

$$\% \text{ Difference} = \frac{\overline{CF} - CF_v}{\overline{CF}} \times 100$$

11.6.2.2 When internal standard calibration is used for PCB congeners, the response factor calculated from the calibration verification standard (RF_v) should not exceed a ±20 percent difference when compared to the mean response factor from the initial calibration. If a calibration approach other than the RSD method has been employed for the initial calibration (e.g., a linear model not through the origin, a non-linear calibration model, etc.), consult Method 8000 for the specifics of calibration verification.

$$\% \text{ Difference} = \frac{\overline{RF} - RF_v}{\overline{RF}} \times 100$$

11.6.2.3 If the calibration does not meet the ±20% limit on the basis of each compound, check the instrument operating conditions, and if necessary, restore them to the original settings, and inject another aliquot of the calibration verification standard. If the response for the analyte is still not within ±20%, then a new initial calibration must be prepared. See Sec. 11.6.6 for a discussion on the effects of a failing calibration verification standard on sample results.

11.6.3 Inject a measured aliquot of the concentrated sample extract. A 2-µL aliquot is suggested, however, other injection volumes may be employed, provided that the analyst can demonstrate adequate sensitivity for the compounds of interest. The same injection volume should be used for both the calibration standards and the sample extracts, unless the analyst can demonstrate acceptable performance using different volumes or conditions. Record the volume injected and the resulting peak size in area units.

11.6.4 Qualitative identifications of target analytes are made by examination of the sample chromatograms, as described in Sec. 11.7.

11.6.5 Quantitative results are determined for each identified analyte (Aroclors or congeners), using the procedures described in Secs. 11.8 and 11.9 for either the internal or the external calibration procedure (Method 8000). If the responses in the sample chromatogram exceed the calibration range of the system, dilute the extract and reanalyze. Peak height measurements are recommended over peak area when overlapping peaks cause errors in area integration.

11.6.6 Each sample analysis employing external standard calibration must be bracketed with an acceptable initial calibration, calibration verification standard(s) (each 12-hr analytical shift), or calibration standards interspersed within the samples. The results from these bracketing standards must meet the calibration verification criteria in Sec. 11.6.2.

Multi-level standards (mixtures or multi-component analytes) are highly recommended to ensure that detector response remains stable for all analytes over the calibration range.

When a calibration verification standard fails to meet the QC criteria, all samples that were injected after the last standard that met the QC criteria must be evaluated to prevent misquantitations and possible false negative results, and reinjection of the sample extracts may be required. More frequent analyses of standards will minimize the number of sample extracts that would have to be reinjected if the QC limits are violated for the standard analysis.

However, if the standard analyzed after a group of samples exhibits a response for an analyte that is above the acceptance limit, i.e., >20%, and the analyte was not detected in the specific samples analyzed during the analytical shift, then the extracts for those samples do not need to be reanalyzed, since the verification standard has demonstrated that the analyte would have been detected if it were present. In contrast, if an analyte

above the QC limits was detected in a sample extract, then reinjection is necessary to ensure accurate quantitation.

If an analyte was not detected in the sample and the standard response is more than 20% below the initial calibration response, then reinjection is necessary. The purpose of this reinjection is to ensure that the analyte could be detected, if present, despite the change in the detector response, e.g., to protect against a false negative result.

11.6.7 Sample injections may continue for as long as the calibration verification standards and standards interspersed with the samples meet instrument QC requirements. It is *recommended* that standards be analyzed after every 10 samples (*required* after every 20 samples and at the end of a set) to minimize the number of samples that must be re-injected when the standards fail the QC limits. The sequence ends when the set of samples has been injected or when qualitative or quantitative QC criteria are exceeded.

11.6.8 The use of internal standard calibration techniques does not require that all sample results be bracketed with calibration verification standards. However, when internal standard calibration is used, the retention times of the internal standards and the area responses of the internal standards should be checked for each analysis. Retention time shifts of more than 30 sec from the retention time of the most recent calibration standard and/or changes in internal standard areas of more than -50 to +100% are cause for concern and must be investigated.

11.6.9 If the peak response is less than 2.5 times the baseline noise level, the validity of the quantitative result may be questionable. The analyst should consult with the source of the sample to determine whether further concentration of the sample is warranted.

11.6.10 Use the calibration standards analyzed during the sequence to evaluate retention time stability. If any of the standards fall outside their daily retention time windows, the system is out of control. Determine the cause of the problem and correct it.

11.6.11 If compound identification or quantitation is precluded due to interferences (e.g., broad, rounded peaks or ill-defined baselines are present), corrective action is warranted. Cleanup of the extract or replacement of the capillary column or detector may be necessary. The analyst may begin by rerunning the sample on another instrument to determine if the problem results from analytical hardware or the sample matrix. Refer to Method 3600 for the procedures to be followed in sample cleanup.

11.7 Qualitative identification

The identification of PCBs as either Aroclors or congeners using this method with an electron capture detector is based on agreement between the retention times of peaks in the sample chromatogram with the retention time windows established through the analysis of standards of the target analytes. See Method 8000 for information on the establishment of retention time windows.

Tentative identification of an analyte occurs when a peak from a sample extract falls within the established retention time window for a specific target analyte. Confirmation is necessary when the sample composition is not well characterized. See Method 8000 for information on confirmation of tentative identifications. See Sec. 11.11 of this procedure for information on the use of GC/MS as a confirmation technique.

When results are confirmed using a second GC column of dissimilar stationary phase, the analyst should check the agreement between the quantitative results on both columns once the identification has been confirmed. See Method 8000 for a discussion of such a comparison and appropriate data reporting approaches.

11.7.1 When simultaneous analyses are performed from a single injection (the dual-column GC configuration described in Sec. 11.3), it is not practical to designate one column as the analytical (primary) column and the other as the confirmation column. Since the calibration standards are analyzed on both columns, both columns must meet the calibration acceptance criteria. If the retention times of the peaks on both columns fall within the retention time windows on the respective columns, then the target analyte identification has been confirmed.

11.7.2 The results of a single column/single injection analysis may be confirmed, if necessary, on a second, dissimilar, GC column. In order to be used for confirmation, retention time windows must have been established for the second GC column. In addition, the analyst must demonstrate the sensitivity of the second column analysis. This demonstration must include the analysis of a standard of the target analyte at a concentration at least as low as the concentration estimated from the primary analysis. That standard may be either the individual congeners, individual Aroclor or the Aroclor 1016/1260 mixture.

11.7.3 When samples are analyzed from a source known to contain specific Aroclors, the results from a single-column analysis may be confirmed on the basis of a clearly recognizable Aroclor pattern. This approach should not be attempted for samples from unknown or unfamiliar sources or for samples that appear to contain mixtures of Aroclors. In order to employ this approach, the analyst must document:

- The peaks that were evaluated when comparing the sample chromatogram and the Aroclor standard.
- The absence of major peaks representing any other Aroclor.
- The source-specific information indicating that Aroclors are anticipated in the sample (e.g., historical data, generator knowledge, etc.).

This information should either be provided to the data user or maintained by the laboratory.

11.7.4 See Sec. 11.11 for information on GC/MS confirmation.

11.8 Quantitation of PCBs as congeners

11.8.1 The quantitation of PCB congeners is accomplished by the comparison of the sample chromatogram to those of the PCB congener standards, using the internal standard technique (see Method 8000). Calculate the concentration of each congener.

11.8.2 Depending on project requirements, the PCB congener results may be reported as congeners, or may be summed and reported as total PCBs. The analyst should use caution when using the congener method for quantitation when regulatory requirements are based on Aroclor concentrations. See Sec. 11.9.3.

11.8.3 The analytical procedures for these 19 congeners may be appropriate for the analysis of other congeners not specifically included in this method and may be used

as a template for the development of such a procedure. However, all 209 PCB congeners cannot be separated using the GC columns and procedures described in this method. If this procedure is expanded to encompass other congeners, then the analyst must either document the resolution of the congeners in question or establish procedures for reporting the results of coeluting congeners that are appropriate for the intended application.

11.9 Quantitation of PCBs as Aroclors

The quantitation of PCB residues as Aroclors is accomplished by comparison of the sample chromatogram to that of the most similar Aroclor standard. A choice must be made as to which Aroclor is most similar to that of the residue and whether that standard is truly representative of the PCBs in the sample.

11.9.1 Use the individual Aroclor standards (not the 1016/1260 mixtures) to determine the pattern of peaks on Aroclors 1221, 1232, 1242, 1248, and 1254. The patterns for Aroclors 1016 and 1260 will be evident in the mixed calibration standards.

11.9.2 Once the Aroclor pattern has been identified, compare the responses of 3 to 5 major peaks in the single-point calibration standard for that Aroclor with the peaks observed in the sample extract. The amount of Aroclor is calculated using the individual calibration factor for each of the 3 to 5 characteristic peaks chosen in Sec. 11.4.6.1. and the calibration model (linear or non-linear) established from the multi-point calibration of the 1016/1260 mixture. Non-linear calibration may result in different models for each selected peak. A concentration is determined using each of the characteristic peaks, using the individual calibration factor calculated for that peak in Sec. 11.4.8, and then those 3 to 5 concentrations are averaged to determine the concentration of that Aroclor.

11.9.3 Weathering of PCBs in the environment and changes resulting from waste treatment processes may alter the PCBs to the point that the pattern of a specific Aroclor is no longer recognizable. Samples containing more than one Aroclor present similar problems. If the purpose of the analysis is <u>not</u> regulatory compliance monitoring on the basis of Aroclor concentrations, then it may be more appropriate to perform the analyses using the PCB congener approach described in this method. If results in terms of Aroclors <u>are</u> required, then the quantitation as Aroclors may be performed by measuring the total area of the PCB pattern and quantitating on the basis of the Aroclor standard that is most similar to the sample. Any peaks that are not identifiable as PCBs on the basis of retention times should be subtracted from the total area. When quantitation is performed in this manner, the problems should be fully described for the data user and the specific procedures employed by the analyst should be thoroughly documented.

11.10 Confirmation

Tentative identification of an analyte occurs when a peak from a sample extract falls within the daily retention time window. Confirmation is necessary when the sample composition is not well characterized. Confirmatory techniques such as gas chromatography with a dissimilar column or a mass spectrometer should be used. See Method 8000 for information on confirmation of tentative identifications.

When results are confirmed using a second GC column of dissimilar stationary phase, the analyst should check the agreement between the quantitative results on both columns once the identification has been confirmed. See Method 8000 for a discussion of such a comparison and appropriate data reporting approaches.

When the dual-column approach is employed, the target phenols are identified and confirmed when they meet the identification criteria on both columns.

11.11 GC/MS confirmation

GC/MS confirmation may be used in conjunction with either single-or dual-column analysis if the concentration is sufficient for detection by GC/MS.

11.11.1 Full-scan quadrupole GC/MS will normally require a higher concentration of the analyte of interest than full-scan ion trap or selected ion monitoring techniques. The concentrations will be instrument-dependent, but values for full-scan quadrupole GC/MS may be as high as 10 ng/µL in the final extract, while ion trap or SIM may only be a concentration of 1 ng/µL.

11.11.2 The GC/MS must be calibrated for the target analytes when it is used for quantitative analysis. If GC/MS is used only for confirmation of the identification of the target analytes, then the analyst must demonstrate that those PCBs identified by GC/ECD can be confirmed by GC/MS. This demonstration may be accomplished by analyzing a single-point standard containing the analytes of interest at or below the concentrations reported in the GC/ECD analysis. When using SIM techniques, the ions and retention times should be characteristic of the Aroclors to be confirmed.

11.11.3 GC/MS confirmation should be accomplished by analyzing the same extract used for GC/ECD analysis and the extract of the associated blank.

11.12 GC/AED confirmation by Method 8085 may be used in conjunction with either single-column or dual-column analysis if the concentration is sufficient for detection by GC/AED.

11.13 Chromatographic system maintenance as corrective action

When system performance does not meet the established QC requirements, corrective action is required, and may include one or more of the following.

11.13.1 Splitter connections

For dual columns which are connected using a press-fit Y-shaped glass splitter or a Y-shaped fused-silica connector, clean and deactivate the splitter port insert or replace with a cleaned and deactivated splitter. Break off the first few centimeters (up to 30 cm) of the injection port side of the column. Remove the columns and solvent backflush according to the manufacturer's instructions. If these procedures fail to eliminate the degradation problem, it may be necessary to deactivate the metal injector body and/or replace the columns.

11.13.2 Metal injector body

Turn off the oven and remove the analytical columns when the oven has cooled. Remove the glass injection port insert (instruments with on-column injection). Lower the injection port temperature to room temperature. Inspect the injection port and remove any noticeable foreign material.

11.13.2.1 Place a beaker beneath the injector port inside the oven. Using a wash bottle, rinse the entire inside of the injector port with acetone and then rinse it with toluene, catching the rinsate in the beaker.

11.13.2.2 Consult the manufacturer's instructions regarding deactivating the injector port body. Glass injection port liners may need deactivation with a silanizing solution containing dimethyldichlorosilane. After all metal surfaces inside the injector body have been thoroughly coated with the deactivation solution, rinse the injector body with toluene, methanol, acetone, then hexane. Reassemble the injector and replace the columns.

11.13.3 Column rinsing

Rinse the column with several column volumes of an appropriate solvent. Both polar and nonpolar solvents are recommended. Depending on the nature of the sample residues expected, the first rinse might be water, followed by methanol and acetone. Methylene chloride is a good final rinse and in some cases may be the only solvent necessary. Fill the column with methylene chloride and allow it to stand flooded overnight to allow materials within the stationary phase to migrate into the solvent. Afterwards, flush the column with fresh methylene chloride, drain the column, and dry it at room temperature with a stream of ultrapure nitrogen.

12.0 DATA ANALYSIS AND CALCULATIONS

See Secs. 11.6 through 11.9 for information regarding data analysis and calculations.

13.0 METHOD PERFORMANCE

13.1 Performance data and related information are provided in SW-846 methods only as examples and guidance. The data do not represent required performance goals for users of the methods. Instead, performance criteria should be developed on a project-specific basis, and the laboratory should establish in-house QC performance criteria for the application of this method. These performance data are not intended to be and must not be used as absolute QC acceptance criteria for purposes of laboratory accreditation.

13.2 The accuracy and precision obtainable with this method depend on the sample matrix, sample preparation technique, optional cleanup techniques, and calibration procedures used. Table 8 provides single laboratory recovery data for Aroclors spiked into clay and soil and extracted with automated Soxhlet. Table 9 provides multiple laboratory data on the precision and accuracy for Aroclors spiked into soil and extracted by automated Soxhlet. These data are provided for guidance purposes only.

13.3 During method performance studies, the concentrations determined as Aroclors were higher than those obtained using the congener method for the limited set of congeners listed in Sec. 1.1. In certain soils, interference prevented the measurement of congener 66. Recoveries of congeners from environmental reference materials ranged from 51 - 66% of the certified Aroclor values, illustrating the potential difficulties in using congener analysis to demonstrate compliance with Aroclor-based regulatory limits. These data are provided for guidance purposes only.

13.4 Tables 10 and 11 contain laboratory performance data for several PCB congeners using supercritical fluid extraction (Method 3562) on an HP 7680 to extract solid samples, including soils, sewage sludge, and fish tissue. Seven replicate extractions were performed on each sample. The method was performed using a variable restrictor and solid trapping material (Florisil). These data are provided for guidance purposes only. Sample analysis was performed by GC/ECD. The following solid samples were used for this study:

13.4.1 Two field-contaminated certified reference materials were extracted by a single laboratory. One of the materials (EC-5) was a lake sediment from Environment Canada. The other material (EC-1) was soil from a dump site and was provided by the National Science and Engineering Research Council of Canada. The average recoveries for EC-5 are based on the certified value for that sample. The average recoveries for EC-1 are based on the certified value of the samples or a Soxhlet value, if a certified value was unavailable for a specific analyte. These data are provided for guidance purposes only.

13.4.2 Four certified reference materials were extracted by two independent laboratories. The materials included a marine sediment from NIST (SRM 1941), a fish tissue from NIST (SRM 2974), a sewage sludge from BCR European Union (CRM 392), and a soil sample from BCR European Union (CRM 481). The average recoveries were based on the certified value of the samples or a Soxhlet value, if a certified value was unavailable for a specific analyte. These data are provided for guidance purposes only.

13.4.3 A weathered sediment sample from Michigan (Saginaw Bay) was extracted by a single laboratory. Soxhlet extractions were carried out on this sample and the SFE recovery is relative to that for each congener. The average recoveries were based on the certified value of the samples. Additional data are shown in the tables for some congeners for which no certified values were available. These data are provided for guidance purposes only.

13.5 Tables 12 through 14 contain single laboratory recovery data for Aroclor 1254 using solid-phase extraction (Method 3535). Recovery data at 2, 10, and 100 µg/L are presented. Results represent three replicate solid-phase extractions of spiked wastewaters. Two different wastewaters from each wastewater type were spiked. All of the extractions were performed using 90-mm C_{18} disks. These data are provided for guidance purposes only.

13.6 Single-laboratory data were developed for PCBs extracted by pressurized fluid extraction (Method 3545) from sewage sludge, a river sediment standard reference material (SRM 1939), and a certified soil reference material (CRM911-050). Certified values were available for five PCB congeners for the sewage sludge and for four congeners in SRM 1939. The soil reference material was certified for Aroclor 1254. All pressurized fluid extractions were conducted using hexane:acetone (1:1), at 100 °C, 1300-1500 psi, and a 5-min static extraction. Extracts were analyzed by GC/ECD. The data are presented in Tables 15 through 17 and are reported in detail in Reference 13. These data are provided for guidance purposes only.

13.7 Single-laboratory accuracy data were obtained for PCBs extracted by microwave extraction (Method 3546) from three reference materials, EC-1, EC-2, and EC-3, from Environment Canada. Natural soils, glass fiber, and sand samples were also used as matrices that were spiked with PCBs. Concentrations varied between 0.2 and 10 µg/g (total PCBs). All samples were extracted using 1:1 hexane:acetone. Extracts were analyzed by GC/ECD. Method blanks, spikes and spike duplicates were included for the low concentration spikes; matrix spikes were included for all other concentrations. The data are presented in Tables 18 through 20 and are reported in detail in Reference 14. These data are provided for guidance purposes only.

14.0 POLLUTION PREVENTION

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity and/or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operations. The EPA has established a preferred hierarchy of

environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best option.

14.2 For information about pollution prevention that may be applicable to laboratories and research institutions consult *Less is Better: Laboratory Chemical management for Waste Reduction* available from the American Chemical Society, Department of Government Relations and Science Policy, 1155 16th Street, NW, Washington, DC, 20036, http://www.acs.org.

15.0 WASTE MANAGEMENT

The Environmental Protection Agency requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. The Agency urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any sewer discharge permits and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management, consult *The Waste Management Manual for Laboratory Personnel* available from the American Chemical Society at the address listed in Sec. 14.2.

16.0 REFERENCES

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12. C. Markell, "3M Data Submission to EPA," letter to B. Lesnik, June 27, 1995.

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17.0 TABLES, DIAGRAMS, FLOW CHARTS, AND VALIDATION DATA

The following pages contain the tables and figures referenced by this method.

TABLE 1

SUGGESTED GC OPERATING CONDITIONS FOR PCBs AS AROCLORS
SINGLE-COLUMN ANALYSIS

Narrow-bore columns

Narrow-bore Column 1 -- 30-m x 0.25 or 0.32-mm ID fused-silica capillary column chemically bonded with SE-54 (DB-5 or equivalent), 1 µm film thickness.

Carrier gas (He)	16 psi
Injector temperature	225 °C
Detector temperature	300 °C
Initial temperature	100 °C, hold 2 min
Temperature program	100 °C to 160 °C at 15 °C/min, followed by 160 °C to 270 °C at 5 °C/min
Final temperature	270 °C

Narrow-bore Column 2 -- 30-m x 0.25-mm ID fused-silica capillary column chemically bonded with 35 percent phenyl methylpolysiloxane (DB-608, SPB-608, or equivalent) 25 µm coating thickness, 1 µm film thickness

Carrier gas (N_2)	20 psi
Injector temperature	225 °C
Detector temperature	300 °C
Initial temperature	160 °C, hold 2 min
Temperature program	160 °C to 290 °C at 5 °C/min
Final temperature	290 °C, hold 1 min

Wide-bore columns

Wide-bore Column 1 -- 30-m x 0.53-mm ID fused-silica capillary column chemically bonded with 35 percent phenyl methylpolysiloxane (DB-608, SPB-608, RTx-35, or equivalent), 0.5 µm or 0.83 µm film thickness.

Wide-bore Column 2 -- 30-m x 0.53-mm ID fused-silica capillary column chemically bonded with 14% cyanopropylmethylpolysiloxane (DB-1701, or equivalent), 1.0 µm film thickness.

Carrier gas (He)	5-7 mL/min
Makeup gas (argon/methane [P-5 or P-10] or N_2)	30 mL/min
Injector temperature	250 °C
Detector temperature	290 °C
Initial temperature	150 °C, hold 0.5 min
Temperature program	150 °C to 270 °C at 5 °C/min
Final temperature	270 °C, hold 10 min

TABLE 1
(continued)

SUGGESTED GC OPERATING CONDITIONS FOR PCBs AS AROCLORS
SINGLE-COLUMN ANALYSIS

Wide-bore Columns (continued)

Wide-bore Column 3 -- 30-m x 0.53-mm ID fused-silica capillary column chemically bonded with SE-54 (DB-5, SPB-5, RTx-5, or equivalent), 1.5 µm film thickness.

Carrier gas (He)	6 mL/min
Makeup gas (argon/methane [P-5 or P-10] or N_2)	30 mL/min
Injector temperature	205 °C
Detector temperature	290 °C
Initial temperature	140 °C, hold 2 min
Temperature program	140 °C to 240 °C at 10 °C/min, hold 5 min at 240 °C, 240 °C to 265 °C at 5 °C/min
Final temperature	265 °C, hold 18 min

TABLE 2

SUGGESTED GC OPERATING CONDITIONS FOR PCBs AS AROCLORS
FOR THE DUAL-COLUMN METHOD OF ANALYSIS

Column 1 -- DB-1701 or equivalent, 30-m x 0.53-mm ID, 1.0 µm film thickness.

Column 2 -- DB-5 or equivalent, 30-m x 0.53-mm ID, 1.5 µm film thickness.

Carrier gas (He) flow rate	6 mL/min
Makeup gas (N_2) flow rate	20 mL/min
Temperature program	0.5 min hold 150 °C to 190 °C, at 12 °C/min, 2 min hold 190 °C to 275 °C, at 4 °C/min, 10 min hold
Injector temperature	250 °C
Detector temperature	320 °C
Injection volume	2 µL
Solvent	Hexane
Type of injector	Flash vaporization
Detector type	Dual ECD
Range	10
Attenuation	64 (DB-1701)/64 (DB-5)
Type of splitter	J&W Scientific press-fit Y-shaped inlet splitter

TABLE 3
(continued)

TABLE 3

EXAMPLE RETENTION TIMES OF AROCLORS
ON THE DB-5 COLUMN[a], DUAL-COLUMN ANALYSIS

Peak No.	Aroclor 1016	Aroclor 1221	Aroclor 1232	Aroclor 1242	Aroclor 1248	Aroclor 1254	Aroclor 1260
1		5.85	5.85				
2		7.63	7.64	7.57			
3	8.41	8.43	8.43	8.37			
4	8.77	8.77	8.78	8.73			
5	8.98	8.99	9.00	8.94	8.95		
6	9.71			9.66			
7	10.49	10.50	10.50	10.44	10.45		
8	10.58	10.59	10.59	10.53			
9	10.90		10.91	10.86	10.85		
10	11.23	11.24	11.24	11.18	11.18		
11	11.88		11.90	11.84	11.85		
12	11.99		12.00	11.95			
13	12.27	12.29	12.29	12.24	12.24		
14	12.66	12.68	12.69	12.64	12.64		
15	12.98	12.99	13.00	12.95	12.95		
16	13.18		13.19	13.14	13.15		
17	13.61		13.63	13.58	13.58	13.59	13.59
18	13.80		13.82	13.77	13.77	13.78	
19	13.96		13.97	13.93	13.93	13.90	
20	14.48		14.50	14.46	14.45	14.46	
21	14.63		14.64	14.60	14.60		
22	14.99		15.02	14.98	14.97	14.98	
23	15.35		15.36	15.32	15.31	15.32	
24	16.01			15.96			
25			16.14	16.08	16.08	16.10	
26	16.27		16.29	16.26	16.24	16.25	16.26
27						16.53	
28			17.04		16.99	16.96	16.97
29			17.22	17.19	17.19	17.19	17.21
30			17.46	17.43	17.43	17.44	
31					17.69	17.69	
32				17.92	17.91	17.91	
33				18.16	18.14	18.14	
34			18.41	18.37	18.36	18.36	18.37
35			18.58	18.56	18.55	18.55	
36							18.68
37			18.83	18.80	18.78	18.78	18.79
38			19.33	19.30	19.29	19.29	19.29

TABLE 3
(continued)

Peak No.	Aroclor 1016	Aroclor 1221	Aroclor 1232	Aroclor 1242	Aroclor 1248	Aroclor 1254	Aroclor 1260
39						19.48	19.48
40						19.81	19.80
41			20.03	19.97	19.92	19.92	
42						20.28	20.28
43					20.46	20.45	
44						20.57	20.57
45				20.85	20.83	20.83	20.83
46			21.18	21.14	21.12	20.98	
47					21.36	21.38	21.38
48						21.78	21.78
49				22.08	22.05	22.04	22.03
50						22.38	22.37
51						22.74	22.73
52						22.96	22.95
53						23.23	23.23
54							23.42
55						23.75	23.73
56						23.99	23.97
57							24.16
58						24.27	
59							24.45
60						24.61	24.62
61						24.93	24.91
62							25.44
63						26.22	26.19
64							26.52
65							26.75
66							27.41
67							28.07
68							28.35
69							29.00

[a] GC operating conditions are given in Table 2. All retention times in minutes and are provided for illustrative purposes only. Each laboratory must determine retention times and retention time windows for their specific application of the method.

[b] The peaks listed in this table are sequentially numbered in elution order for illustrative purposes only and are not isomer numbers.

TABLE 4

EXAMPLE RETENTION TIMES OF AROCLORS
ON THE DB-1701 COLUMN[a], DUAL-COLUMN ANALYSIS

Peak No.	Aroclor 1016	Aroclor 1221	Aroclor 1232	Aroclor 1242	Aroclor 1248	Aroclor 1254	Aroclor 1260
1		4.45	4.45				
2		5.38					
3		5.78					
4		5.86	5.86				
5	6.33	6.34	6.34	6.28			
6	6.78	6.78	6.79	6.72			
7	6.96	6.96	6.96	6.90	6.91		
8	7.64			7.59			
9	8.23	8.23	8.23	8.15	8.16		
10	8.62	8.63	8.63	8.57			
11	8.88		8.89	8.83	8.83		
12	9.05	9.06	9.06	8.99	8.99		
13	9.46		9.47	9.40	9.41		
14	9.77	9.79	9.78	9.71	9.71		
15	10.27	10.29	10.29	10.21	10.21		
16	10.64	10.65	10.66	10.59	10.59		
17				10.96	10.95	10.95	
18	11.01		11.02	11.02	11.03		
19	11.09		11.10				
20	11.98		11.99	11.94	11.93	11.93	
21	12.39		12.39	12.33	12.33	12.33	
22			12.77	12.71	12.69		
23	12.92			12.94	12.93		
24	12.99		13.00	13.09	13.09	13.10	
25	13.14		13.16				
26						13.24	
27	13.49		13.49	13.44	13.44		
28	13.58		13.61	13.54	13.54	13.51	13.52
29				13.67		13.68	
30			14.08	14.03	14.03	14.03	14.02
31			14.30	14.26	14.24	14.24	14.25
32					14.39	14.36	
33			14.49	14.46	14.46		
34						14.56	14.56
35					15.10	15.10	
36			15.38	15.33	15.32	15.32	
37			15.65	15.62	15.62	15.61	16.61
38			15.78	15.74	15.74	15.74	15.79
39			16.13	16.10	16.10	16.08	
40							16.19
41						16.34	16.34

TABLE 4
(continued)

Peak No.	Aroclor 1016	Aroclor 1221	Aroclor 1232	Aroclor 1242	Aroclor 1248	Aroclor 1254	Aroclor 1260
42						16.44	16.45
43						16.55	
44			16.77	16.73	16.74	16.77	16.77
45			17.13	17.09	17.07	17.07	17.08
46						17.29	17.31
47				17.46	17.44	17.43	17.43
48				17.69	17.69	17.68	17.68
49					18.19	18.17	18.18
50				18.48	18.49	18.42	18.40
51						18.59	
52						18.86	18.86
53				19.13	19.13	19.10	19.09
54						19.42	19.43
55						19.55	19.59
56						20.20	20.21
57						20.34	
58							20.43
59					20.57	20.55	
60						20.62	20.66
61						20.88	20.87
62							21.03
63						21.53	21.53
64						21.83	21.81
65						23.31	23.27
66							23.85
67							24.11
68							24.46
69							24.59
70							24.87
71							25.85
72							27.05
73							27.72

[a] GC operating conditions are given in Table 2. All retention times are in minutes and are provided for illustrative purposes only. Each laboratory must determine retention times and retention time windows for their specific application of the method.

[b] The peaks listed in this table are sequentially numbered in elution order for illustrative purposes only and are not isomer numbers.

TABLE 5

EXAMPLE RETENTION TIMES OF PEAKS DIAGNOSTIC OF PCBs
ON A 0.53-mm ID COLUMNS DURING SINGLE-COLUMN ANALYSIS

Peak No.[a]	RT on DB-608[b]	RT on DB-1701[b]	Aroclor[c]
I	4.90	4.66	1221
II	7.15	6.96	1221, 1232, 1248
III	7.89	7.65	1061, 1221, 1232, 1242
IV	9.38	9.00	1016, 1232, 1242, 1248
V	10.69	10.54	1016, 1232, 1242
VI	14.24	14.12	1248, 1254
VII	14.81	14.77	1254
VIII	16.71	16.38	1254
IX	19.27	18.95	1254, 1260
X	21.22	21.23	1260
XI	22.89	22.46	1260

[a]Peaks are sequentially numbered in elution order and are not isomer numbers

[b]Temperature program: T_i = 150 °C, hold 30 sec; 5 °C/min to 275 °C.

[c]Underline indicates the largest peak in the pattern for that Aroclor

All retention times are in minutes and are provided for illustrative purposes only. Each laboratory must determine retention times and retention time windows for their specific application of the method.

TABLE 6

SPECIFIC PCB CONGENERS THAT ARE MAJOR COMPONENTS IN COMMON AROCLORS

		Aroclor						
Congener	IUPAC Number	1016	1221	1232	1242	1248	1254	1260
Biphenyl	--		X					
2-CB	1	X	X	X	X			
2,3-DCB	5	X	X	X	X	X		
3,4-DCB	12	X		X	X	X		
2,4,4'-TCB	28*	X		X	X	X	X	
2,2',3,5'-TCB	44			X	X	X	X	X
2,3',4,4'-TCB	66*					X	X	X
2,3,3',4',6-PCB	110						X	
2,3',4,4',5-PCB	118*						X	X
2,2',4,4',5,5'-HCB	153							X
2,2',3,4,4',5'-HCB	138							X
2,2',3,4,4',5,5'-HpCB	180							X
2,2',3,3',4,4',5-HpCB	170							X

*Apparent co-elution of: 28 with 31 (2,4',5-trichlorobiphenyl)
66 with 95 (2,2',3,5',6-pentachlorobiphenyl)
118 with 149 (2,2',3,4',5',6-hexachlorobiphenyl)

This table is not intended to illustrate all of the congeners that may be present in a given Aroclor, but rather to illustrate the major congener components.

TABLE 7
EXAMPLE RETENTION TIMES OF PCB CONGENERS ON THE DB-5 WIDE-BORE COLUMN

IUPAC Number	Retention Time (min)
1	6.52
5	10.07
18	11.62
31	13.43
52	14.75
44	15.51
66	17.20
101	18.08
87	19.11
110	19.45
151	19.87
153	21.30
138	21.79
141	22.34
187	22.89
183	23.09
180	24.87
170	25.93
206	30.70
209 (internal standard)	32.63

All data are provided for illustrative purposes only. Each laboratory must determine retention times and retention time windows for their specific application of the method.

TABLE 8

EXAMPLE SINGLE-LABORATORY RECOVERY DATA FOR THE EXTRACTION OF PCBs FROM CLAY AND SOIL BY AUTOMATED SOXHLET (METHOD 3541)[a]

Matrix	Aroclor	Spike Level (ppm)	Trial	Percent Recovery[b]
Clay	1254	5	1	87
			2	93
			3	94
			4	99
			5	79
			6	28
Clay	1254	50	1	65
			2	72
			3	97
			4	80
			5	50
			6	59
Clay	1260	5	1	87
			2	75
			3	61
			4	94
			5	97
			6	113
Clay	1260	50	1	74
			2	70
			3	92
			4	89
			5	90
			6	67

TABLE 8
(continued)

Matrix	Aroclor	Spike Level (ppm)	Trial	Percent Recovery[b]
Soil	1254	5	1	70
			2	89
			3	92
			4	83
			5	63
Soil	1254	50	1	84
			2	78
			3	92
			4	67
			5	82
			6	62
Soil	1260	5	1	84
			2	83
			3	82
			4	96
			5	94
			6	94
			7	98
Soil	1260	50	1	77
			2	69
			3	93
			4	82
			5	83
			6	76

[a]The operating conditions for the automated Soxhlet
Immersion time: 60 min
Reflux time: 60 min

[b]Multiple results from two different extractors

Data are taken from Reference 9
These data are provided for guidance purposes only.

TABLE 9

EXAMPLE MULTIPLE-LABORATORY PRECISION AND ACCURACY DATA
FOR THE EXTRACTION OF PCBs FROM SPIKED SOIL
BY AUTOMATED SOXHLET (METHOD 3541)

		Percent Recovery at Aroclor 1254 Spike Concentration (µg/kg)			Percent Recovery at Aroclor 1260 Spike Concentration (µg/kg)			Mean Recovery
		5	50	500	5	50	500	All Levels
	n	3	3		3	3		12
Lab 1	Mean	101.2	74.0		83.9	78.5		84.4
	S. D.	34.9	41.8		7.4	7.4		26.0
	n		6	6		6	6	24
Lab 2	Mean		56.5	66.9		70.1	74.5	67.0
	S. D.		7.0	15.4		14.5	10.3	13.3
	n	3	3		3	3		12
Lab 3	Mean	72.8	63.3		70.6	57.2		66.0
	S. D.	10.8	8.3		2.5	5.6		9.1
	n	6	6		6	6		24
Lab 4	Mean	112.6	144.3		100.3	84.8		110.5
	S. D.	18.2	30.4		13.3	3.8		28.5
	n		3	3		3	3	12
Lab 5	Mean		97.1	80.1		79.5	77.0	83.5
	S. D.		8.7	5.1		3.1	9.4	10.3
	n	2	3		3	4		12
Lab 6	Mean	140.9	127.7		138.7	105.9		125.4
	S. D.	4.3	15.5		15.5	7.9		18.4
	n	3	3		3	3		12
Lab 7	Mean	100.1	123.4		82.1	94.1		99.9
	S. D.	17.9	14.6		7.9	5.2		19.0
	n	3	3		3	3		12
Lab 8	Mean	65.0	38.3		92.8	51.9		62.0
	S. D.	16.0	21.9		36.5	12.8		29.1
	n	20	30	9	21	31	9	120
All	Mean	98.8	92.5	71.3	95.5	78.6	75.3	87.6
Labs	S. D.	28.7	42.9	14.1	25.3	18.0	9.5	29.7

Data are taken from Reference 7
These data are provided for guidance purposes only.

TABLE 10

EXAMPLE PERCENT RECOVERY (BIAS) OF PCBs IN VARIOUS SOILS
USING SUPERCRITICAL FLUID EXTRACTION (METHOD 3562)

PCB No.[a]	EC-1 Dump Site Soil Low #1	SRM 1941 Marine Sediment Low #2	EC-5 Lake Sediment Low #3	CRM 481[b] European Soil High #1	Saginaw Bay Sediment High #2	CRM 392 Sewage Sludge High #3	SRM 2974 Fish Tissue Mussel Low #4	Congener Mean
28	148.4	63.3	147.7	67.3	114.7	89.2	101.7	104.6
52	88.5	106.6	115.8	84.5	111.1	96.2	131.4	104.9
101	93.3	91.2	100.2	84.5	111.5	93.9	133.2	101.1
149	92.6	105.1	101.5	73.2	111.2		69.4	92.2
118	89.9	66.1	108.9	82.1	110.8	73.5	82.7	87.7
153	90.8	65.1	95.1	82.8	118.6	97.3	107.5	94.0
105[b]	89.1	72.6	96.6	83.4	111.8		79.4	88.8
138	90.1	57.4	97.9	76.9	126.9		73.1	87.1
128	90.8	69.9	101.2	65.9	87.6		62.5	79.7
156[b]	90.6	88.9	94.3	85.2	101.1		59.3	86.6
180	92.4	142.4	93.3	82.2	109.2	100.5	65.7	98.0
170	91.3	101.1	95.2	80.5			33.0	81.8
Matrix Mean	95.7	85.8	104.0	79.0	108.7	91.8	83.2	92.2

[a] Congeners which are either certified or have had Soxhlet confirmation.
[b] Congener 105 was not resolved from congener 132 and congener 156 was not resolved from congener 171 by the GC method used for samples EC-1 and EC-5.

TABLE 11

PRECISION (AS %RSD) OF PCBs EXTRACTED USING SUPERCRITICAL FLUID EXTRACTION (METHOD 3562)

PCB No.[a]	EC-1 Dump Site Soil Low #1	SRM 1941 Marine Sediment Low #2	EC-5 Lake Sediment Low #3	CRM 481 European Soil High #1	Saginaw Bay Sediment High #2	CRM 392 Sewage Sludge High #3	SRM 2974 Fish Tissue Mussel Low #4	Congener Mean
28	11.5	1.5	3.8	5.6	2.4	1.9	2.7	4.2
52	9.1	3.3	3.9	5.4	2.2	2.9	3.1	4.3
101	9.1	2.9	2.8	4.9	1.4	5.2	2.9	4.2
149	7.1	0.7	3.8	3.9	3.4		2.2	3.0
118	9.8	1.9	4.5	5.4	2.0	3.3	2.4	4.2
153	8.4	1.5	3.0	4.3	4.3	9.5	3.0	4.9
105[b]	6.6	3.7	2.7	4.3	2.7		2.5	3.2
138	9.2	1.8	3.1	4.7	2.3		2.9	3.4
128	6.0	5.3	3.3	4.9	2.8		3.3	3.7
156[b]	8.3	0.0	5.1	4.5	1.9		3.8	3.4
180	8.0	1.3	3.6	4.3	3.1	9.6	2.7	4.7
170	5.7	2.3	3.6	3.9	2.3		4.0	3.1
Matrix Mean	8.2	2.2	3.6	4.7	2.6	2.7	3.0	3.8

[a] Congeners which are either certified or have had Soxhlet confirmation.
[b] Congener 105 was not resolved from congener 132 and congener 156 was not resolved from congener 171 by the GC method used for samples EC-1 and EC-5.

These data are provided for guidance purposes only.

TABLE 12

EXAMPLE SINGLE-LABORATORY RECOVERY DATA FOR SOLID-PHASE EXTRACTION (METHOD 3535) OF AROCLOR 1254 FROM WASTEWATER MATRICES SPIKED AT 2 µg/L

Wastewater Type	Mean Conc. (µg/L)	Percent Recovery	Std. Dev. (µg/L)	RSD (%)
Chemical Industry	2.4	120	0.41	17.2
Chemical Industry	0.6	28	0.03	5.4
Paper Industry	3.0	150	0.56	18.5
Paper Industry	2.3	115	0.08	3.7
Pharmaceutical Industry	1.5	76	0.03	1.7
Pharmaceutical Industry	1.0	51	0.03	2.9
Refuse	0.5	27	0.04	6.7
Refuse	0.6	31	0.10	16.0
POTW	1.9	96	0.15	7.8
POTW	2.1	105	0.04	1.8

Results represent three replicate solid-phase extractions of spiked wastewaters. Two different wastewaters from each wastewater type were spiked. All extractions were performed using 90-mm C_{18} extraction disks.
These data are provided for guidance purposes only.

TABLE 13

EXAMPLE SINGLE-LABORATORY RECOVERY DATA FOR SOLID-PHASE EXTRACTION (METHOD 3535) OF AROCLOR 1254 FROM WASTEWATER MATRICES SPIKED AT 10 µg/L

Wastewater Type	Mean Conc. (µg/L)	Percent Recovery	Std. Dev. (µg/L)	RSD (%)
Chemical Industry	8.8	88	1.07	12.2
Chemical Industry	8.1	81	0.06	0.7
Paper Industry	8.9	89	0.71	7.9
Paper Industry	10.1	101	0.15	1.4
Pharmaceutical Industry	9.2	92	0.24	2.6
Pharmaceutical Industry	8.4	84	0.17	2.0
Refuse	8.8	88	0.49	5.6
Refuse	8.0	80	1.44	18.0
POTW	9.5	82	0.17	2.1
POTW	8.2	82	0.17	2.1

Results represent three replicate solid-phase extractions of spiked wastewaters. Two different wastewaters from each wastewater type were spiked. All extractions were performed using 90-mm C_{18} extraction disks.
These data are provided for guidance purposes only.

TABLE 14

EXAMPLE SINGLE-LABORATORY RECOVERY DATA
FOR SOLID-PHASE EXTRACTION (METHOD 3535) OF AROCLOR 1254
FROM WASTEWATER MATRICES SPIKED AT 100 µg/L

Wastewater Type	Mean Conc. (µg/L)	Percent Recovery	Std. Dev. (µg/L)	RSD (%)
Chemical Industry	81.7	82	1.46	1.8
Chemical Industry	89.7	90	0.66	0.7
Paper Industry	73.7	74	3.94	5.3
Paper Industry	95.3	95	1.89	2.0
Pharmaceutical Industry	86.4	86	1.95	2.3
Pharmaceutical Industry	79.2	79	3.92	4.9
Refuse	85.7	86	1.59	1.9
Refuse	71.5	72	1.61	2.2
POTW	87.8	88	1.76	2.0
POTW	80.6	81	0.40	0.5

Results represent three replicate solid-phase extractions of spiked wastewaters. Two different wastewaters from each wastewater type were spiked. All extractions were performed using 90-mm C_{18} extraction disks.
These data are provided for guidance purposes only.

TABLE 15

EXAMPLE SINGLE-LABORATORY PCB CONGENER DATA
FROM A SEWAGE SLUDGE SAMPLE EXTRACTED BY
PRESSURIZED FLUID EXTRACTION (METHOD 3545)

PCB No.	Mean Recovery (%)	%RSD	Certified Value (µg/kg)
52	114	4.7	163
101	143	7.4	161
138	110	3.9	193
153	110	5.8	198
180	160	7.5	207

Percent recoveries are the mean of six replicate extractions.

Data are taken from Reference 13.
These data are provided for guidance purposes only.

TABLE 16

EXAMPLE SINGLE-LABORATORY PCB CONGENER DATA
FROM A RIVER SEDIMENT REFERENCE MATERIAL
EXTRACTED BY PRESSURIZED FLUID EXTRACTION (METHOD 3545)

PCB No.	Mean Recovery (%)	%RSD	Certified Value (µg/kg)
101	89	3.7	780
138	122	2.3	570
153	62	4.1	370
180	112	5.9	180

Percent recoveries are the mean of six replicate extractions.
The river sediment reference material was SRM 1939.

Data are taken from Reference 13.
These data are provided for guidance purposes only.

TABLE 17

EXAMPLE SINGLE-LABORATORY AROCLOR 1254 DATA
FROM A SOIL REFERENCE MATERIAL
EXTRACTED BY PRESSURIZED FLUID EXTRACTION (METHOD 3545)

Replicate Extraction	Aroclor 1254 Concentration (µg/kg)
1	1290
2	1370
3	1280
4	1370
Mean	1330
%RSD	3.5%
Certified value	1340
Mean recovery (%)	99%

Data are taken from Reference 13.
These data are provided for guidance purposes only.

TABLE 18

EXAMPLE SINGLE-LABORATORY PCB HOMOLOGUE DATA BY MICROWAVE EXTRACTION (METHOD 3546) FROM A CERTIFIED GREAT LAKE SEDIMENT MATERIAL (EC-2)

	Microwave Extraction			Soxhlet Extraction		
PCB homologue	µg/kg	Peaks[a]	% RSD	µg/kg	Peaks[a]	% RSD
Trichlorobiphenyl	130	4	21.8	100	4	14.6
Tetrachlorobiphenyl	400	10	13.2	390	20	10.2
Pentachlorobiphenyl	310	9	1.9	300	9	8.7
Hexachlorobiphenyl	120	3	0.0	110	3	9.1

[a] Number of PCB peaks detected
Cl_3 to Cl_{10} homologues analyzed
n=3
Data are taken from Reference 14. These data are provided for guidance purposes only.

TABLE 19

EXAMPLE SINGLE-LABORATORY PCB HOMOLOGUE DATA BY MICROWAVE EXTRACTION (METHOD 3546) FROM A CERTIFIED HARBOR SEDIMENT MATERIAL (SRM-1944)

	Microwave Extraction			Soxhlet Extraction		
PCB homologue	µg/kg	Peaks[a]	% RSD	µg/kg	Peaks[a]	% RSD
Trichlorobiphenyl	450	8	10.1	360	6	5.8
Tetrachlorobiphenyl	580	12	3.9	580	11	6.0
Pentachlorobiphenyl	330	9	6.1	330	9	7.9
Hexachlorobiphenyl	260	3	12.4	240	3	5.1
Heptachlorobiphenyl	60	2	43.8	80	2	27.3

[a] Number of PCB peaks detected
Cl_3 to Cl_{10} homologues analyzed
n=3
Data are taken from Reference 14. These data are provided for guidance purposes only.

TABLE 20

EXAMPLE SINGLE-LABORATORY PCB DATA BY MICROWAVE EXTRACTION (METHOD 3546) FROM CERTIFIED GREAT LAKE SEDIMENT MATERIALS

Sediment	Total Aroclor Concentration (µg/kg)	Standard Deviation (µg/kg)	RSD (%)	n	Certified Value (µg/kg)
EC-1	1850	0.07	3.78	3	2000 ± 54
EC-2	1430	0.09	6.60	4	1160 ± 70
EC-3	670	0.02	3.12	3	660 ± 54

Sample size = 2 g extracted into a final volume of 4 mL

EC-2 and EC-3 certified values were only provisional values at the time the work was conducted. The data presented herein were part of the validation data package used to confirm the certified values.

Data are taken from Reference 14.
These data are provided for guidance purposes only.

FIGURE 1. Example GC/ECD chromatogram of the Aroclor 1016/1260 mixture analyzed on a Rtx-5/HP-608 column pair connected to separate injectors. The top trace is the Rtx-5 column (30-m x 0.53-mm ID, 1.5-µm film thickness) and the bottom trace is the HP-608 column (30-m x 0.53-mm ID, 0.5-µm film thickness). Temperature program: 150 °C (1.0 min hold) to 280 °C (17 min hold) at 8 °C/min.

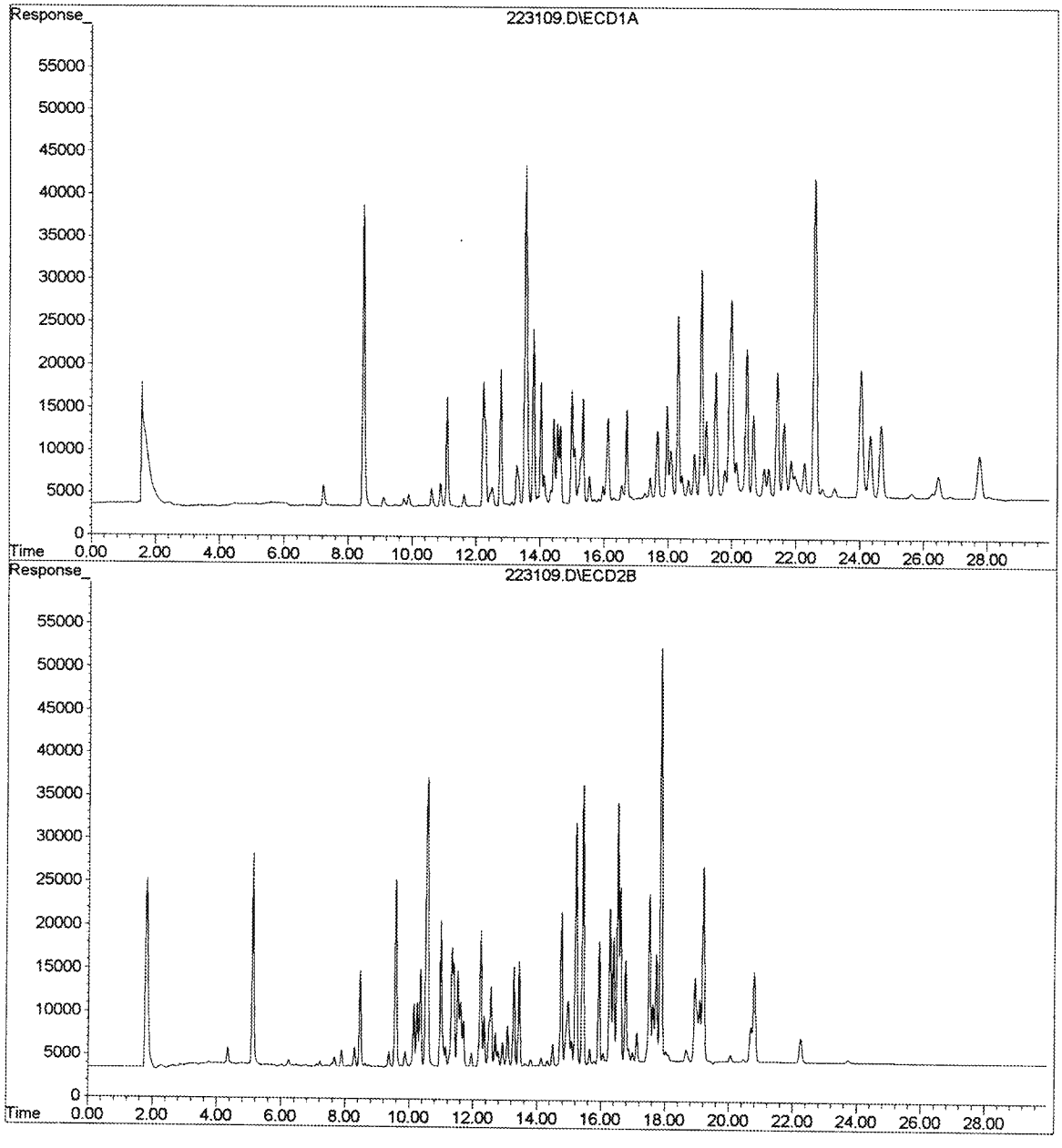


FIGURE 2. Example GC/ECD chromatogram of Aroclor 1221 analyzed on a Rtx-5/HP-608 column pair connected to separate injectors. The top trace is the Rtx-5 column (30-m x 0.53-mm ID, 1.5-µm film thickness) and the bottom trace is the HP-608 column (30-m x 0.53-mm ID, 0.5-µm film thickness). Temperature program: 150 °C (1.0 min hold) to 280 °C (17 min hold) at 8 °C/min.

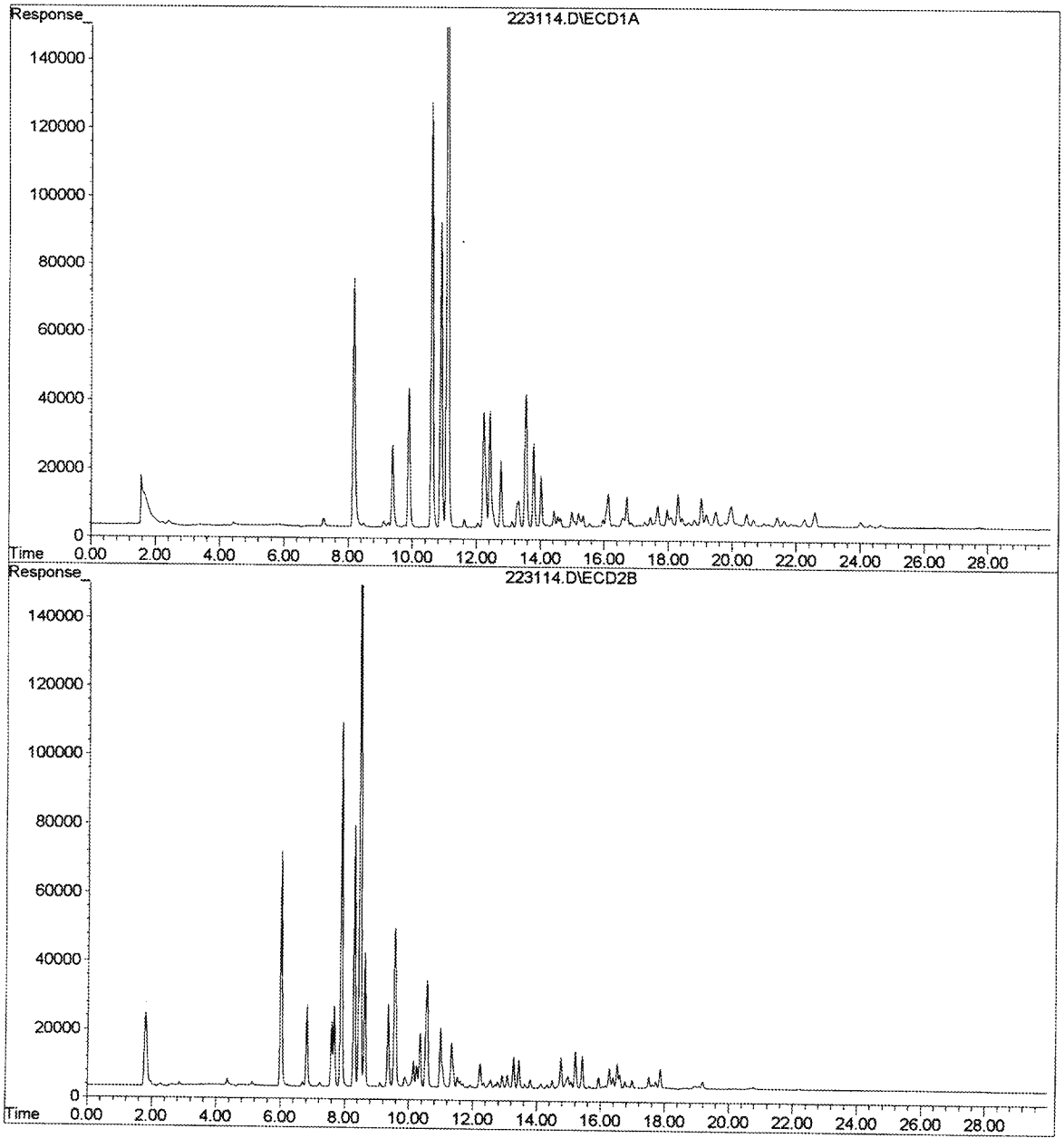


FIGURE 3. Example GC/ECD chromatogram of Aroclor 1232 analyzed on a Rtx-5/HP-608 column pair connected to separate injectors. The top trace is the Rtx-5 column (30-m x 0.53-mm ID, 1.5-µm film thickness) and the bottom trace is the HP-608 column (30-m x 0.53-mm ID, 0.5-µm film thickness). Temperature program: 150 °C (1.0 min hold) to 280 °C (17 min hold) at 8 °C/min.

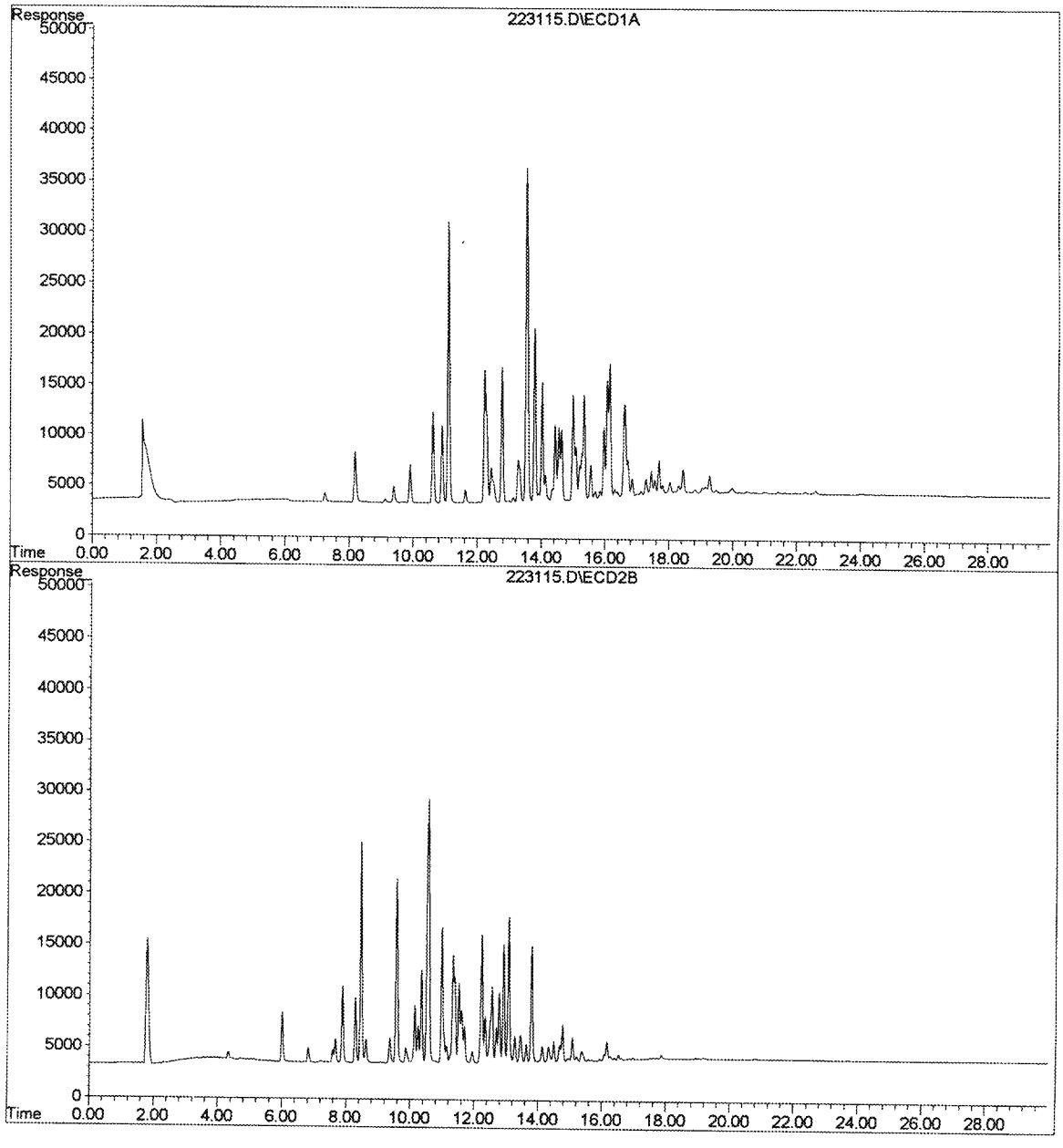


FIGURE 4. Example GC/ECD chromatogram of Aroclor 1242 analyzed on a Rtx-5/HP-608 column pair connected to separate injectors. The top trace is the Rtx-5 column (30-m x 0.53-mm ID, 1.5-µm film thickness) and the bottom trace is the HP-608 column (30-m x 0.53-mm ID, 0.5-µm film thickness). Temperature program: 150 °C (1.0 min hold) to 280 °C (17 min hold) at 8 °C/min.

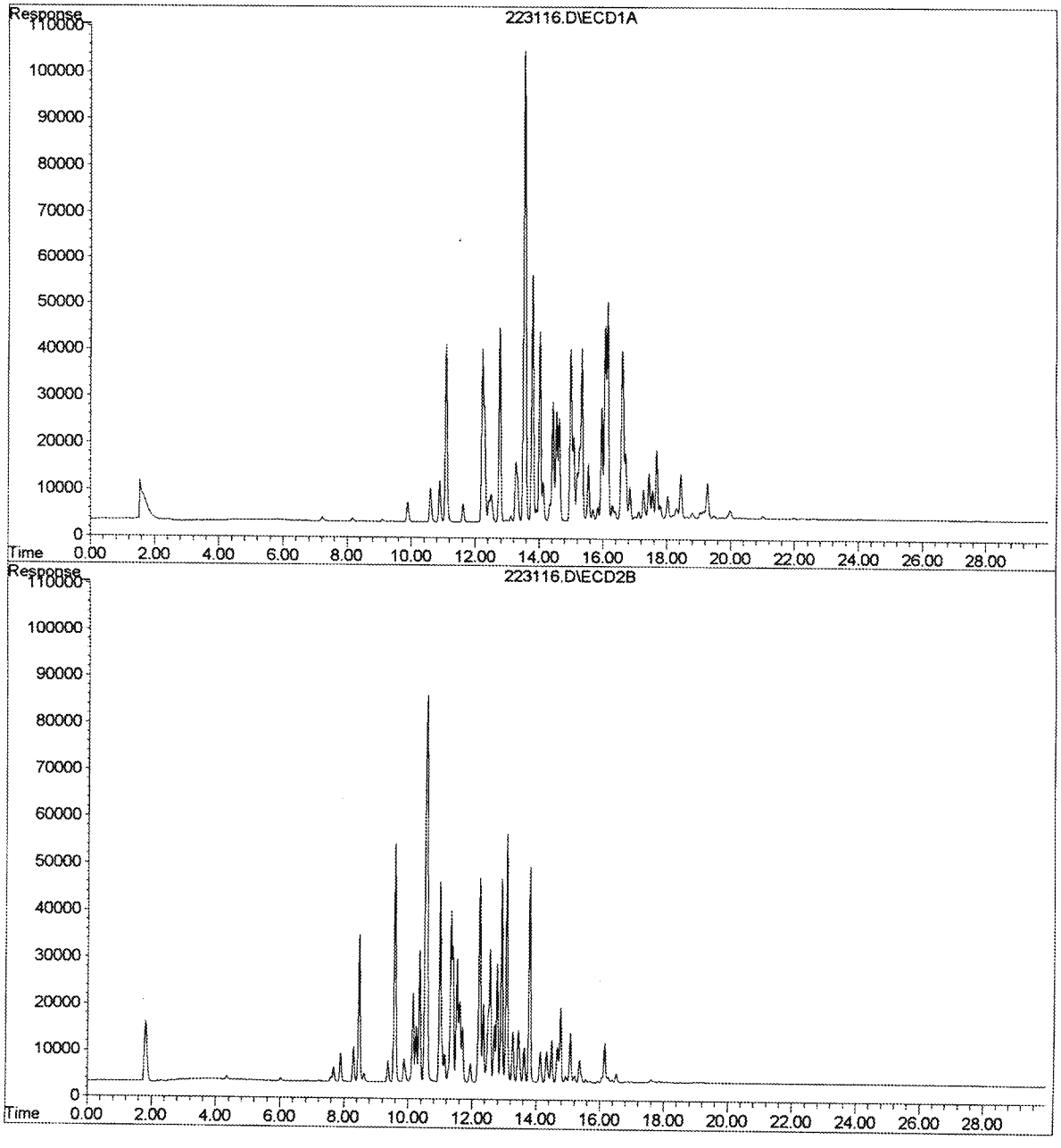


FIGURE 5. Example GC/ECD chromatogram of Aroclor 1248 analyzed on a Rtx-5/HP-608 column pair connected to separate injectors. The top trace is the Rtx-5 column (30-m x 0.53-mm ID, 1.5-µm film thickness) and the bottom trace is the HP-608 column (30-m x 0.53-mm ID, 0.5-µm film thickness). Temperature program: 150 °C (1.0 min hold) to 280 °C (17 min hold) at 8 °C/min.

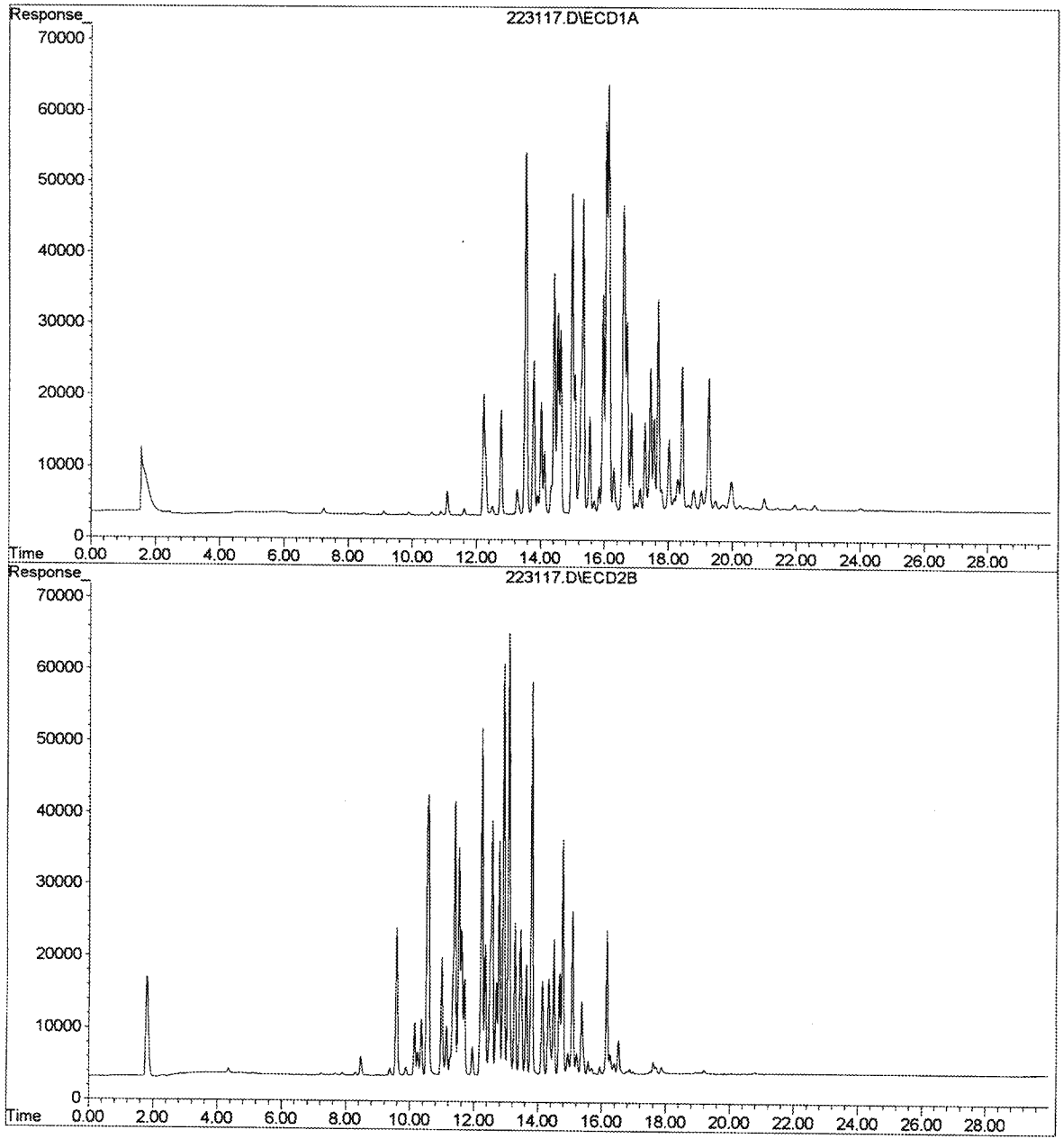
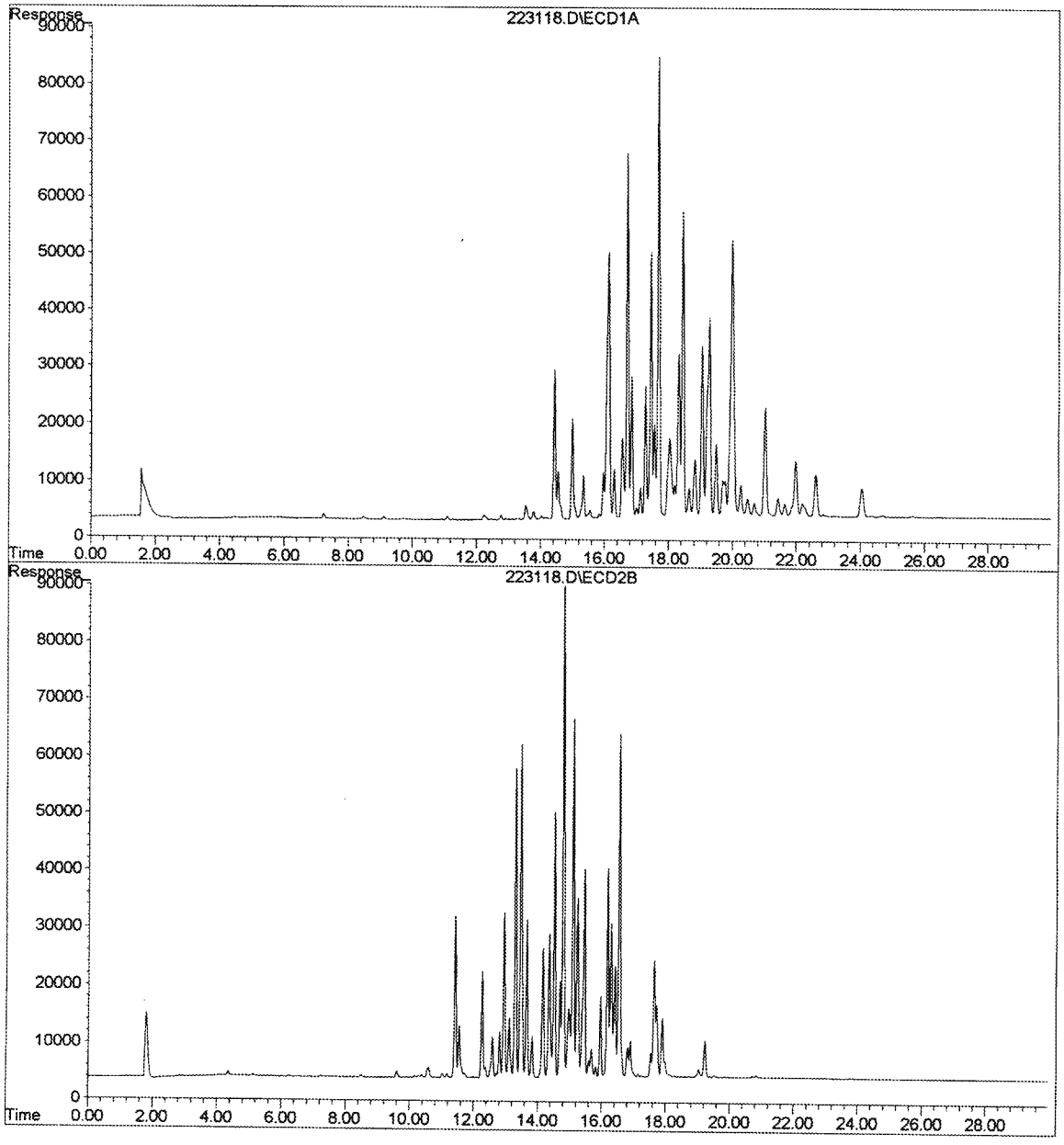


FIGURE 6. Example GC/ECD chromatogram of Aroclor 1254 analyzed on a Rtx-5/HP-608 column pair connected to separate injectors. The top trace is the Rtx-5 column (30-m x 0.53-mm ID, 1.5-µm film thickness) and the bottom trace is the HP-608 column (30-m x 0.53-mm ID, 0.5-µm film thickness). Temperature program: 150 °C (1.0 min hold) to 280 °C (17 min hold) at 8 °C/min.



Appendix D

Example Chain of Custody and Blank ALS Standard Chain of Custody

Field Chain-of-Custody Record

Page 1 of 3

39147

1703194

Ship To: ALS Environmental
4388 Glendale Milford Rd.
Cincinnati, Ohio 45242
Phone: (513) 733-5336
Fax: (513) 733-5347

REGULAR Status ☐ RUSH Status ☒ RESULTS REQUIRED BY: (Date) Thursday 3/9/2017
CONTACT ALS ENVIRONMENTAL PRIOR TO SENDING SAMPLES

OH VAP: ☐ YES ☐ NO BUSTR: ☐ YES ☐ NO

Date: 3/6/2017 Purchase Order No.:

Company Name: Fulcrum Environmental Project No.: 17-2070

Address: 406 N 2nd St Sampling Site: Sky Valley Edu Center

City: Yakima State: WA Zip: 98901

Person to Contact: Ryan Mathews + Levi Wyatt Billing Address (if different):

Email Address: rmathews@fulcrum.net
lwyatte@fulcrum.net

Telephone ( ): 509-574-0839

Alternate Contact:

ALS Lab ID	Sample ID / Description	Date	Time	Preservation Key #	Sample Type / Matrix Key Abbr.	# of Sample Containers	TO-10a	ANALYSIS REQUESTED
01	030617-01 Room F-A / Annex Bld Room F	3/6/17					X	
02	030617-02 Girl Stg-A / Storage Room Girls Locker Rm						X	
03	030617-03 Girl LR-A / Girls Locker Room						X	
04	030617-04 Sm Gym-A / Small Gym						X	
05	030617-05 Elec Sm Gym A / Electrical Rm @ Small Gym						X	
06	030617-06 Gathering Rm A / Gathering Room						X	
07	030617-07 CTE-A / CTE Bldg Classroom						X	
08	030617-08 Large Gym Electrical Room / Large Gym						X	
09	030617-09 CTE Elec / Electrical Room @ CTE						X	
10	030617-10 Pod Mezz / West Pod Mezzanine						X	

Notes:
5 Liters/min for total of 420 mins
Air Samples total volume 2100L

PLEASE HOLD 030617-07 CTE-A & 030617-09 CTE Elec

Preservative Key: 1 - HCl 2 - HNO3 3 - H2SO4 4 - NaOH 5 - Na2S2O3 6 - NaHSO4 7 - NaOH/ZnAcetate 8 - Other 9 - 4°C

Matrix Key: A - Air B - Bulk S - Soil W - Water

Failure to complete all portions of this form may delay analysis. Please fill in this form LEGIBLY.

Relinquished By: (Signature)	Time / Date	Received By: (Signature)	Time / Date
	400 3/6/17		1457 3/7/17
Relinquished By: (Signature)	Time / Date	Received By: (Signature)	Time / Date
Relinquished By: (Signature)	Time / Date	Received By: (Signature)	Time / Date

ALS LAB USE ONLY

COOLER TEMP. 4.0 °C pH ADJUSTMENTS:

COOLING METHOD: NONE (COOLER) WET ICE DRY ICE (ICE PACK)

DELIVERY METHOD: CLIENT DROP BOX FEDEX (UPS) STD MAIL PRTY MAIL ALS COURIER OTHER

CUSTODY SEALS: (NONE) COOLER PACKAGE SAMPLER

EQUIP. RETURNED:

ALS
Ship To: ALS Environmental
4388 Glendale Milford Rd.
Cincinnati, Ohio 45242
Phone: (513) 733-5336
Fax: (513) 733-5347

Field Chain-of-Custody Record

39148

1703194

REGULAR Status ☐ RUSH Status ☒ RESULTS REQUIRED BY: (Date) Thursday 3/9/2017
CONTACT ALS ENVIRONMENTAL PRIOR TO SENDING SAMPLES

OH VAP: ☐ YES ☐ NO BUSTR: ☐ YES ☐ NO

Date: 3/6/2017 Purchase Order No.:

Company Name: Falcrom Environmental Project No.: 17-2070

Address: 406 N 2nd St Sampling Site: Sky Valley Edu Center

City: Yakima State: WA Zip: 98901

Person to Contact: Ryan Mathews & Levi Wyatt Billing Address (if different):

Email Address: rmathews@falcrom.net
lwyatt@falcrom.net

Telephone ( ): 509-574-0839

Alternate Contact:

ALS Lab ID	Sample ID / Description	Date	Time	Preservation Key #	Sample Type / Matrix Key Abbr.	# of Sample Containers	ANALYSIS REQUESTED: EPA SW846-8082
11	030617-11 Elec Sm Gym / metal transformer side	3/6/17					X
12	030617-12 Elec Sm Gym / metal transformer top						X
13	030617-13 Gregga Elec Lg Gym / metal transformer side						X
14	030617-14 Elec Lg Gym / concrete floor						X
15	030617-15 CTE Elec Rm / metal transformer						X
16	030617-16 CTE Elec Rm / concrete floor						X
17	030617-17 West Pod Maze / wood floor						X
18	030617-18 West Pod Maze / metal transformer side						X
19	030617-19 Field Blank						X
20	030617-20 Lab Blank						X

Notes:
wipe samples
10 x 10 template
100 cm² total area

EXAMPLE

Preservation Key: 1 - HCl 2 - HNO3 3 - H2SO4 4 - NaOH 5 - Na2S2O3 6 - NaHSO4 7 - NaOH/ZnAc salts 8 - Other 9 - 4°C

Matrix Key: A - Air B - Bulk S - Soil W - Water

Failure to complete all portions of this form may delay analysis. Please fill in this form LEGIBLY.

Relinquished By: (Signature)	Time / Date	Received By: (Signature)	Time / Date
	9:00 3/6/17		9:57 3/7/17
Relinquished By: (Signature)	Time / Date	Received By: (Signature)	Time / Date
Relinquished By: (Signature)	Time / Date	Received By: (Signature)	Time / Date

ALS LAB USE ONLY

COOLER TEMP: °C pH ADJUSTMENTS:

COOLING METHOD: NONE COOLER WET ICE DRY ICE ICE PACK

DELIVERY METHOD: CLIENT DROP BOX FEDEX UPS
STD MAIL PRTY MAIL ALS COURIER OTHER

CUSTODY SEALS: NONE COOLER PACKAGE SAMPLES

EQUIP. RETURNED:

Ship To: ALS Environmental
4388 Glendale Milford Rd.
Cincinnati, Ohio 45242
Phone: (513) 733-5336
Fax: (513) 733-5347

Field Chain-of-Custody Record

39149

1703194

REGULAR Status ☐ RUSH Status ☒ RESULTS REQUIRED BY: (Date) Thurs 3/9/17
CONTACT ALS ENVIRONMENTAL PRIOR TO SENDING SAMPLES

OH VAP: ☐ YES ☐ NO BUSTR: ☐ YES ☐ NO

Date: 3/6/2017 Purchase Order No.:
Company Name: Tetra Tech Project No.: 17-2070
Address: 406 N 2nd St Sampling Site: Hay Valley Edu Center
City: Yakima State: WA Zip: 98901
Person to Contact: Ryan Mathews Billing Address (if different):
Email Address: rmathews@tetratech.net
Telephone ( ): 509 574 0229
Alternate Contact: Levi Wyatt lwyatt@tetratech.net

ALS Lab ID	Sample ID / Description	Date	Time	Preservation Key #	Sample Type / Matrix Key Abbr.	# of Sample Containers	ANALYSIS REQUESTED: 70 - 109
>1	1703194-21	Field Blank	3/6/17				X
>2	1703194-22	Lab Blank					X

Notes:

Preservation Key: 1 - HCl 2 - HNO_3 3 - H_2SO_4 4 - NaOH 5 - $Na_2S_2O_3$ 6 - NaHSO4 7 - NaOH/ZnAcetate 8 - Other 9 - 4°C

Matrix Key: A - Air B - Bulk S - Soil W - Water

Failure to complete all portions of this form may delay analysis. Please fill in this form LEGIBLY.

Relinquished By: (Signature)	Time / Date	Received By: (Signature)	Time / Date
			907 3/7/17
Relinquished By: (Signature)	Time / Date	Received By: (Signature)	Time / Date
Relinquished By: (Signature)	Time / Date	Received By: (Signature)	Time / Date

ALS LAB USE ONLY

COOLER TEMP: °C pH ADJUSTMENTS:

COOLING METHOD: NONE COOLER WET ICE DRY ICE ICE PACK

DELIVERY METHOD: CLIENT DROP BOX FEDEX UPS
STD MAIL PRTY MAIL ALS COURIER OTHER:

CUSTODY SEALS: NONE COOLER PACKAGE SAMPLES

EQUIP RETURNED:

Field Chain-of-Custody Record

☐ **REGULAR** Status ☐ **RUSH** Status

Page_____of_____

Cooler Temp _____
(Lab only)

Date _____ Purchase Order No. _____

Company Name _____

Address _____

City State Zip

Person to Contact _____

Email Address _____

Telephone ( ) _____

Fax Telephone ( ) _____

Billing Address (if different)

Project No. _____

Sampling Site _____

Date/Time of Collection _____

VAP ☐ Yes ☐ No

Sample Number	Site ID	Date	Time	Lab Sample Number	Preservation	Sample Type	Analysis Requested									No. of Containers

EXAMPLE COC - NOTE: ALS-CINCINNATI PROVIDES THREE PART COC FORMS FOR USE

Notes:

Failure to complete all portions of this form may delay analysis. Please fill in this form *LEGIBLY*.

Relinquished by: (*Signature*)	Time / Date	Received by: (*Signature*)	Time / Date
Relinquished by: (*Signature*)	Time / Date	Received by: (*Signature*)	Time / Date
Relinquished by: (*Signature*)	Time / Date	Received by: (*Signature*)	Time / Date

Ship to: **ALS Environmental**
4388 Glendale - Milford Road
Cincinnati, Ohio 45242
Phone: 513.733.5336
Fax: 513.733.5347

Carrier / Airbill #

Date / Time: